THE LABORATORY BOOK

OF

DAIRY ANALYSIS

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THE LABORATORY BOOK
OF
DAIRY ANALYSIS

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BY THE AUTHOR

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PREFACE TO SECOND EDITION

For some considerable time this little book has been out of print, but the author has now found time to make a few necessary corrections and add information to bring it up to date. A number of new methods have been added, and an endeavour has been made to give information which will aid in the solution of those practical problems which confront the dairy chemist.

October 1912 H. D. R.

PREFACE TO FIRST EDITION

This work is intended to contain working directions for the analysis of milk and dairy-products; the estimation of all constituents of diagnostic value is shortly described in detail, and is in many cases illustrated by photographs of chemists actually carrying out the determination.

A chapter on the application of analysis to the solution of problems usually placed before the chemist is included, and a very short summary of the composition of milk and its products is given.

In the Appendix the composition and preparation of the various solutions is detailed. Tables are given to facilitate the working out of results; these Tables are condensed to occupy one page each, and the saving of time by avoiding the turning over of pages will more than compensate for the slight extra labour due to the condensation.

While not intended to be a complete guide to the analysis of milk, it is hoped that this work will afford assistance to analysts, health officers, dairy students, and those engaged in the supervision of dairies; with this object in view the more simple tests have been described in a manner which will render their working by persons other than chemists possible; it must be remembered, however, that though these methods are easy, they are often fallaciously easy, and lack of chemical training may lead to the making of errors, and the overlooking of important points; no amount of careful following of directions can replace a thorough training in chemical science and manipulation, and though simple tests have a real value as a guide, they have not the reliability of an analysis made by a skilled chemist.

H. D. R.

September 1905

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INTRODUCTION

Milk consists of (1) fat in small globules (Fig. 1) ranging in size from 0.01 mm. in diameter to 0.0016; (2) milk-sugar and (3) various salts in solution in water; (4) casein, combined with lime and phosphoric acid; and (5) albumin in less perfect solution.

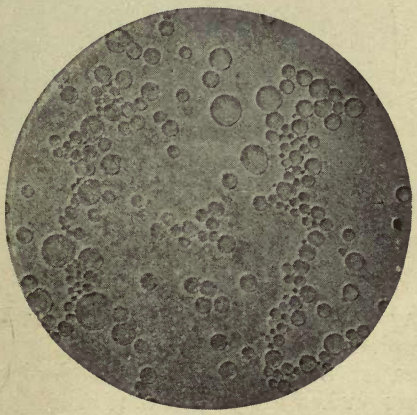


FIG. 1.—Milk (magnified 400 diameters).

There are in addition (6) other compounds in small quantities, including enzymes or natural ferments.

The fat will be treated of in the section on butter; the milk-sugar belongs to the class of carbo-hydrates and crystallises with 1 OH_2, and is one of the hexa-bioses. It rotates the plane of polarisation, its specific rotatory power being 52.5° for the crystallised sugar; and reduces solutions of copper salts.

Casein is a protein belonging to the class of the

phospho-proteins ; it contains carbon, hydrogen, oxygen, nitrogen, sulphur, and phosphorus ; in milk it exists as a salt of lime and soda combined with calcium phosphate ; acids precipitate the free casein if dilute, while strong acids re-dissolve it. Rennet splits casein up into curd, which is a combination of para-casein with the lime and the calcium phosphate of the casein, the

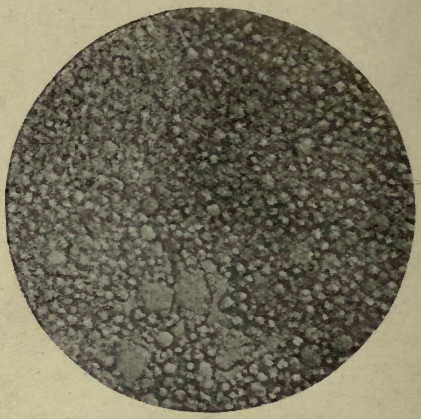


Fig. 2.—Cream (magnified 400 diameters).

soda being split off, and whey protein which is free from phosphorus.

Albumin is a protein which is distinguished by coagulating on heating to 70° C. ; in milk it probably exists as a salt, and this does not coagulate until the milk is acidified. Unaltered albumin is not precipitated by acids.

When micro-organisms act on milk various products are formed ; the most important change is the formation of lactic acid from the sugar, which causes milk to become sour, and curdles it by precipitating the casein.

The fat globules are lighter than the aqueous serum, and they tend to rise. *Cream* (Fig. 2) is the upper portion of milk after standing, and differs from milk practically only in that it contains more fat and pro-

portionately less serum. *Skim-milk* is the milk deprived of the bulk of its cream, and if the separation of cream has been performed in a centrifugal separator it is practically free from fat and contains only the aqueous serum. This is termed *separated* or *machine-skimmed milk*.

When cream (or milk) is suitably agitated for some time, the fat globules coalesce to small granules, and these after working together into a nearly homogeneous mass form *butter*. This is chiefly composed of fat, but contains some water and other constituents of milk. The residue is termed *butter-milk*, which does not differ greatly from skim-milk in composition.

By treating milk with rennet, *curd* is separated; this carries down the bulk of the fat, and after pressing, salting, and ripening, partly by the action of micro-organisms and partly by the action of the natural ferments of milk, it is converted into *cheese*; cheese consists essentially of fat, para-casein, and products derived from the latter together with some water and salts.

The following Table gives the average morning and evening milk for each month, and represents the percentage composition during 1911:

TABLE I

Month	Morning Milk				Evening Milk			
	Sp. gr.	Total Solids	Fat	Solids not Fat	Sp. gr.	Total Solids	Fat	Solids not Fat
		%	%	%		%	%	%
January .	1.0323	12.64	3.68	8.96	1.0321	12.86	3.91	8.95
February .	1.0325	12.55	3.57	8.98	1.0322	12.79	3.83	8.96
March .	1.0324	12.41	3.49	8.92	1.0321	12.66	3.76	8.90
April . .	1.0321	12.31	3.48	8.83	1.0318	12.59	3.77	8.82
May . .	1.0323	12.12	3.26	8.86	1.0319	12.51	3.68	8.83
June . .	1.0323	12.09	3.24	8.85	1.0316	12.42	3.64	8.78
July . .	1.0316	12.17	3.44	8.73	1.0309	12.35	3.74	8.61
August .	1.0309	12.13	3.54	8.59	1.0304	12.42	3.90	8.52
September	1.0311	12.35	3.69	8.66	1.0306	12.68	4.07	8.61
October .	1.0315	12.52	3.76	8.76	1.0312	12.82	4.07	8.75
November.	1.0316	12.65	3.83	8.82	1.0314	12.85	4.05	8.80
December .	1.0318	12.54	3.72	8.82	1.0315	12.69	3.88	8.81

In the Table below is given the average percentage composition of milk and the various products derived from it.

TABLE II

	Water	Fat	Milk Sugar	Proteins	Mineral Matter
MILK . .	**87.25**	**3.75**	**4.75**	**3.40**	**0.75**
Separated Milk	90.40	0.20	4.95	3.57	0.78
Thick Cream .	39.37	56.09	2.29	1.57	0.38
Thin Cream .	67.50	25.67	3.66	2.60	0.57
Fresh Butter .	12.99	85.81	0.37	0.74	0.09
Salt Butter .	13.78	82.97	0.39	0.84	2.02*
Butter-milk .	90.33	0.76	4.78‡	3.33	0.80
Whey . .	93.21	0.30	4.99	0.92	0.58
Curd. . .	49.43	27.38	2.04	20.00	1.15
Cream Cheese .	30.66	62.99	0.26‡	4.94†	1.15
Soft Cheese .	50.04	27.50	?	18.32†	4.12*
Hard Cheese .	33.89	33.00	1.90‡	27.56†	3.65*
Half-skim Cheese	37.35	24.61	?	32.40†	5.65*

* Including added salt.
† Including products of ripening.
‡ Including lactic acid.

CHAPTER II

THE ANALYSIS OF MILK

Preparation of the Sample.—A milk sample conveniently consists of a five-ounce bottle filled nearly full; if the sample is to be representative of a bulk, the milk (whether in a churn, pail, can, or jug) should invariably be well stirred before the sample is taken in order to distribute the cream which always tends to rise to the surface. Samples taken in a dairy or other place for testing on the premises may, however, be taken in cans, and if the sample is one frequently taken from the same source a distinguishing mark may be stamped on the can.

On receipt of the sample in the laboratory it should invariably be stirred before any portion is withdrawn for analysis; violent shaking is to be deprecated, as not only is there a tendency to churn the fat but air bubbles, which do not separate immediately, are included, and prevent accurate measurements, especially of specific gravity. In cold weather samples are often frothy, and while they remain cold the air bubbles separate very slowly. Freshly drawn milk is also frothy, and the fat being in the liquid condition has a lower specific gravity than it has after solidification. If the sample is turning sour there is often difficulty in uniformly distributing the cream, and violent shaking may have to be resorted to; if any of the fat is churned or becomes churned in this operation, the lumps of churned fat should be removed, dried, and the fat extracted with ether and weighed; the total weight of milk is ascertained, and the percentage of churned fat calculated. The remainder of the sample is analysed separately.

Milk which is sour and curdled is mixed by turning

the whole sample into a beaker, and whipping with a brush made of fine wires.

Always determine the specific gravity of every sample if possible.

FIG. 3.—Putting Lactometer into Milk.

Estimation of Specific Gravity by Lactometer.—Nearly fill a cylindrical vessel of depth such that the lactometer will float, and at least $\frac{1}{4}$ inch wider than the lactometer; a glass jar, or tin pot, or even a milk-can serves well. Hold the lactometer at an angle (Fig. 3), and cautiously lower it into the milk,

taking care that no air bubbles are retained in the space between the upper and lower bulbs (*see* Fig. 4); when the upper bulb is partially below the surface, raise to an upright position and immerse the lactometer to the 30°

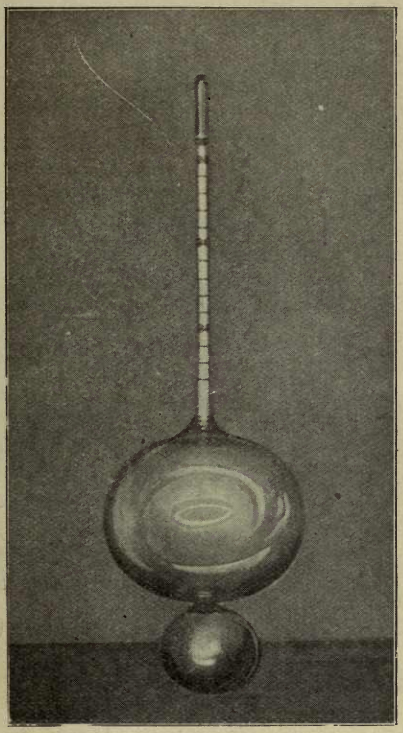


Fig. 4.—Vieth's Lactometer.

mark, and let it find its own level. When steady read off the point where the surface of the milk cuts the stem; this point is not visible, as the milk is drawn up round the stem by capillary attraction (Fig. 5), and must be mentally estimated. Some little practice is required

to do this, and the reading may be obtained by observing the point on the stem to which the milk reaches, and adding a constant amount for the height of the meniscus, usually $\frac{1}{2}$ degree; thus in the figure the true reading of the lactometer is 32.5°, the apparent reading is 32°, and with $\frac{1}{2}$ degree added on the true reading is obtained.

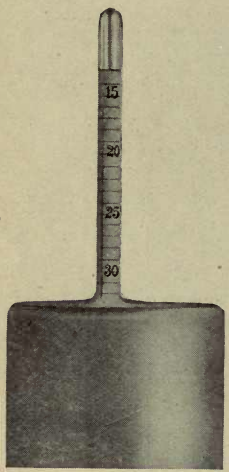


FIG. 5.—Lactometer in Milk.

The jar in the figure has been filled to the brim with milk in order to show clearly the effect of capillary attraction; it is neither necessary nor advisable to do this when testing milk.

Immerse the bulb of a thermometer in the milk, and stir the milk till the temperature is constant; correct the reading to 60° F. by means of the table given on p. 88 or by the milk scale (p. 40). To use the table, find in the top line column the specific gravity (or the nearest figure to the left), and in the left-hand column the temperature; where the lines intersect the corrected specific gravity is given (adding on, if the specific gravity found is not an exact degree, the decimals).

A thermo-lactometer, *i.e.* a lactometer which contains a thermometer, may be used, and the temperature can then be read off from the upper scale at the same time as the specific gravity (Fig. 6).

Never take a specific gravity reading without also noting the temperature and correcting to 60° F.

The lactometer should be checked by the gravimetric method, to make sure that the scale is correct.

A lactometer does not give accurate results if a film of milk is allowed to dry on it; if a sample has been

tested and the lactometer removed, and allowed to stand, it must be washed and dried before being used for another sample.

There is usually little objection, however, to removing

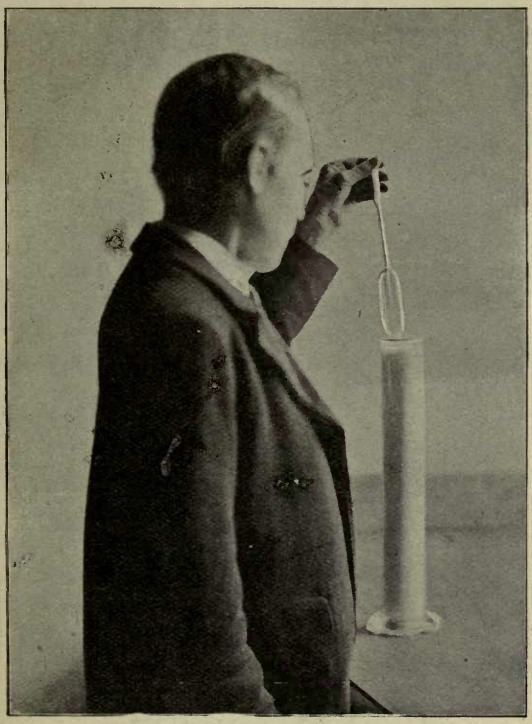


Fig. 6.—Thermo-lactometer.

a lactometer from one sample and, after draining, placing it at once in another sample ; but if the sample is likely to be the subject of legal proceedings or is otherwise important, the lactometer should always be cleaned before testing.

The Gravimetric Estimation of Specific Gravity.—Dry a Sprengel tube (Fig. 7) by washing

with distilled water, alcohol, and ether, and placing in the water-oven; aspirate air through while still hot, cool, and weigh. Fill the tube with distilled water, and place it in a vessel containing water at 15.5° C. till the

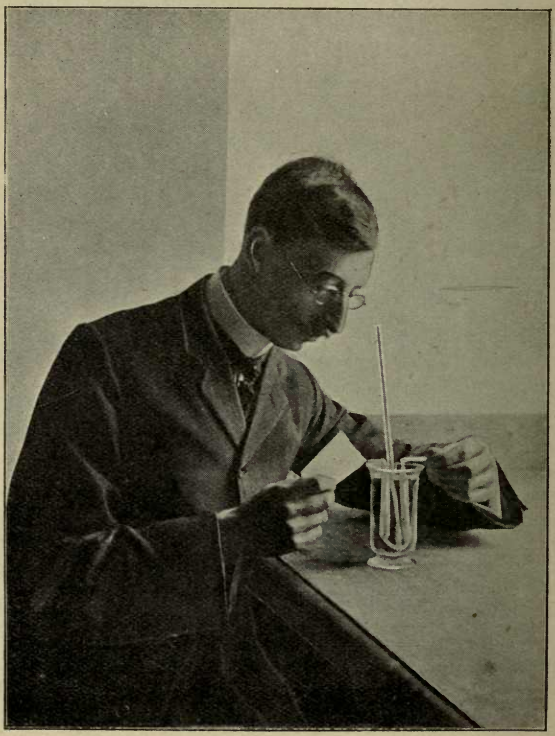


FIG. 7.—Sprengel Tube.

water inside no longer alters in volume; adjust the level of the water accurately to the mark on the wider tube by cautiously applying filter-paper to the narrower end; wipe the outside of the tube dry, and weigh. The difference between the weight of the full

and the empty tube gives its capacity in grammes of water at 15.5°.

Empty the water, and rinse the tube several times with the milk, and then fill it with milk; allow the tube to stand a minute to permit air bubbles to rise, suck or blow these out, and fill the tube completely. Immerse the tube in water at 15.5° C. till the volume ceases to alter, and then wipe dry and weigh as before. The difference between the full and the empty tube gives the weight of milk, and this divided by the capacity in grammes of water at 15.5° gives the specific gravity at 15.5°.

The capacity of the tube once determined remains constant, and it need not therefore be determined every time; the weight of the empty tube should, however, be taken occasionally.

Rise of Specific Gravity of Milk on Standing.—If the milk is freshly drawn or has been recently heated, the fat is in the liquid condition, and its specific gravity is lower than when solid. The solidification of the fat globules takes some time, and twelve to twenty-four hours may elapse before the maximum specific gravity is attained. If the milk is frothy, air bubbles may cause the specific gravity to appear too low.

The maximum specific gravity is taken as the correct figure.

Estimation of Total Solids.—Weigh a basin either of platinum, tantalum, fused silica, or porcelain, preferably $2\frac{3}{4}$ in. wide and flat-bottomed; pipette in 5 c.c. of milk and weigh again; the weighing should be rapid, but the exactitude of weighing need not be more than 2 mg. Place the basin on a water-bath (Fig. 8), and from time to time break the skin with a needle; when apparently dry place in a water-oven (Fig. 9), and continue the drying for four hours, cool in a desiccator, and weigh; replace in the oven for periods of one hour each, cool and weigh, until the loss in one hour is less than 1 mg. The weight of the residue divided by the

weight of milk taken and multiplied by 100 gives the percentage of total solids.

(*Babcock's method*) The basin may be loosely packed with ignited asbestos, if great accuracy is required; (*Stokes' method*) if speed is wanted, a few drops of a 10 per cent. solution of acetic acid in alcohol, or (*Revis' method*) an equal volume of acetone, may be added, and the time of drying can then be curtailed.

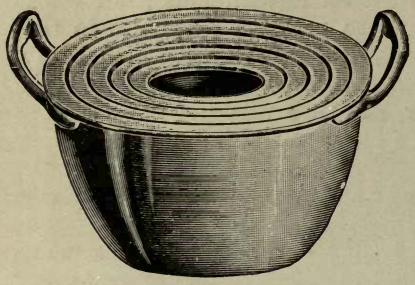


Fig. 8.—Water-bath.

Estimation of Ash.—The total solids are ignited in a muffle furnace (Fig. 10), which should not be allowed to become hotter than a dull red heat; to facilitate burning, upstanding portions of the ash may be broken down by touching with a platinum wire; when all the carbon is burnt away the basin is cooled in a desiccator and weighed. The ignition may be performed over a Bunsen burner (Fig. 11), the point of the flame of which should barely be allowed to touch the bottom of the basin, or an Argand burner may be used; the basin should be covered with a platinum lid. It is very important not to heat the ash to too high a temperature.

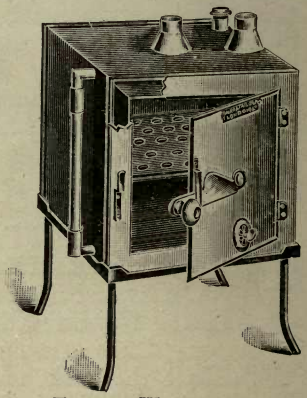


Fig. 9.—Water-oven.

Estimation of Soluble and Insoluble Ash.—Fill the basin with hot water and filter through a

small ash-free filter; wash with hot water. Place the filter and its contents in the basin, and ignite; the

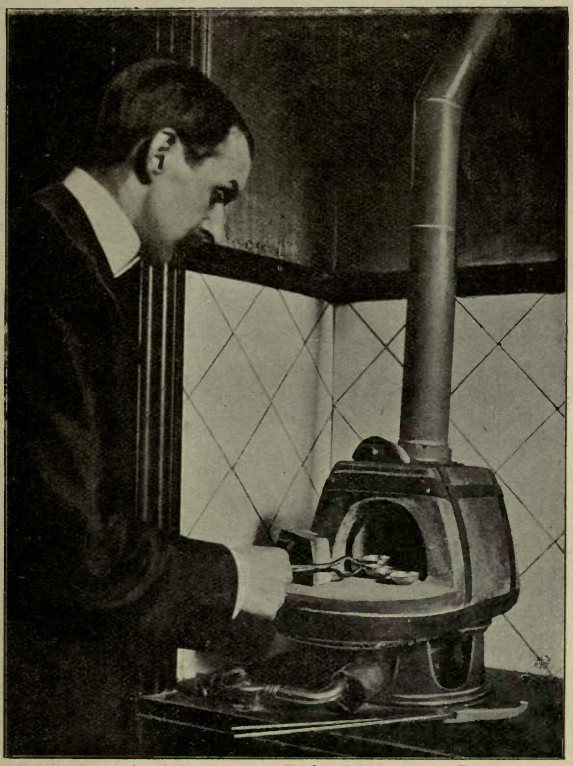


FIG. 10.—Muffle.

residue is the insoluble ash and the soluble ash can be obtained by difference.

Estimation of Alkalinity, Chlorine, Lime, and Phosphoric Acid.—To the filtrate containing the soluble ash add a drop or two of phenolphthalein solution, and titrate with $\frac{N}{10}$ sulphuric acid (*see*

Appendix) till the colour is discharged. Each cubic centimetre of acid indicates 0.0044 gramme CO_2 as

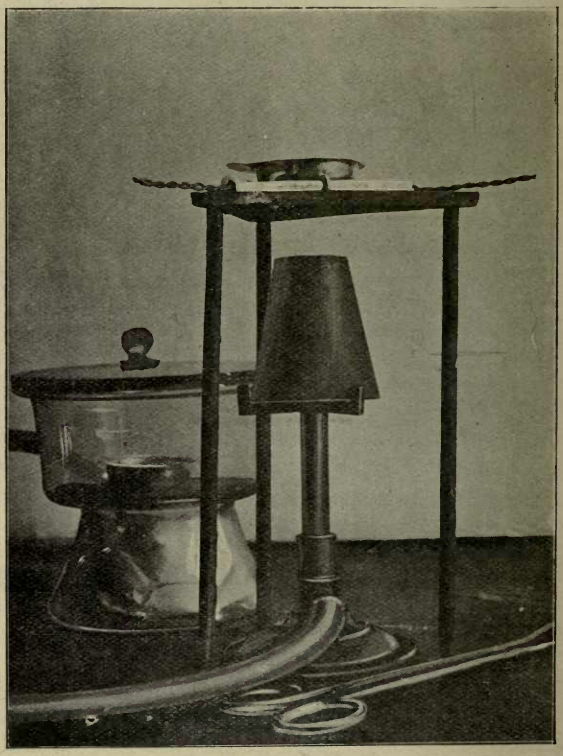


FIG. 11.—Tripod, Bunsen, and Desiccator.

carbonates. [*Note.*—The alkalinity is not all due to carbonates, a little phosphate is present.]

To the solution add a few drops of potassium chromate, and titrate with $\frac{N}{10}$ silver nitrate solution (*see* Appendix) till a red colour just appears. Each cubic centimetre of silver solution indicates 0.00355 gramme

of chlorine. [*Note.*—A little of the chlorine, about 0.01 per cent., is lost on ignition of the total solids.]

For the estimation of lime and phosphoric acid, it is advisable to take another quantity of milk (10 c.c. or 25 c.c. being preferable); this is dried on the water-bath, ignited, the ash dissolved in a little hydrochloric acid, and the solution boiled; after cooling slightly, ammonia is added drop by drop till a permanent turbidity appears, and hydrochloric acid added in quantity sufficient to remove this, excess being avoided. The solution is brought just to the boiling-point, and a saturated solution of ammonium oxalate added drop by drop, so long as a precipitate appears; the solution is kept hot (in a water-oven) for at least two hours, and filtered through a small ash-free filter; the precipitate is transferred to the filter, washed with hot water, and the filter placed in a tared basin, and ignited over a small flame; when the filter-paper is all burnt away, the precipitate is moistened with a solution of ammonium carbonate, dried, and very gently ignited. The precipitate, now converted into calcium carbonate, is weighed, and the weight of lime found by multiplying by 0.56; it is usually slightly grey, and contains traces of iron, which are small enough to be neglected.

To the filtrate is added 5 or 10 c.c. of magnesia mixture (*see* Appendix), and about one-tenth its volume of strong ammonia; and after stirring well the liquid is allowed to stand at least 12 hours; the precipitate is washed by decantation with dilute ammonia, transferred to a filter, and the washing completed; the filter is placed in a weighed basin, and ignited at first gently, and finally very strongly till white; the residue of magnesium pyrophosphate is weighed, and the amount of phosphoric acid (as P_2O_5) found by multiplying by 0.6396.

For the determination of other mineral constituents, works on mineral analysis should be consulted.

The Estimation of Acidity.—Place 10 c.c. of milk in a white porcelain basin (Fig. 12), add 1 c.c. of

phenolphthalein solution (*see* Appendix), and run in from a burette $\frac{N}{10}$ caustic soda, or other alkali solution, strontia being recommended (*see* Appendix), in drops,

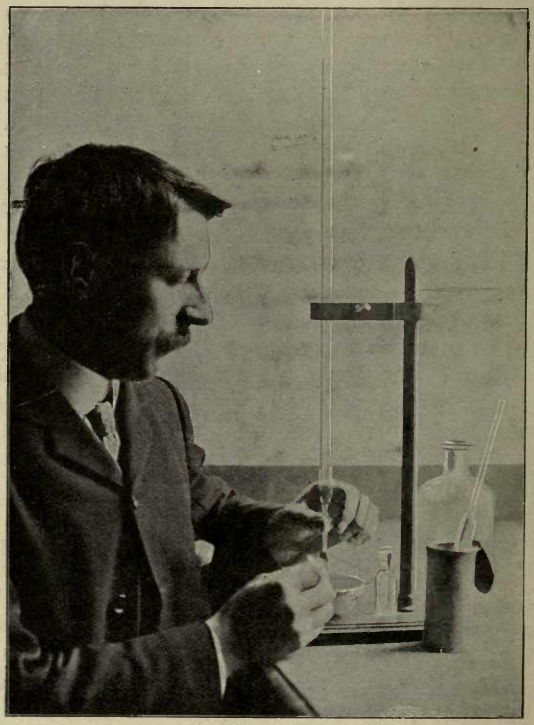


FIG. 12.—Estimation of Acidity.

stirring constantly, till a faint pink colour, equal to that given by adding 1 drop of a 0.01 per cent. solution of rosaniline acetate in 96 per cent. alcohol to 11 c.c. of the same milk, is produced; each $\frac{1}{10}$ c.c. of $\frac{N}{10}$ alkali solution represents 1° acidity, and the degrees

multiplied by 0.009 will give the acidity in percentage of lactic acid.

If greater accuracy is required 50 c.c. may be taken, and 5 c.c. of phenolphthalein solution added; each $\frac{1}{2}$ c.c. will then represent 1° acidity.

The Volumetric Estimation of Fat.—Gerber's method of fat estimation consists in reading the volume of fat brought into the graduated neck of a bottle by

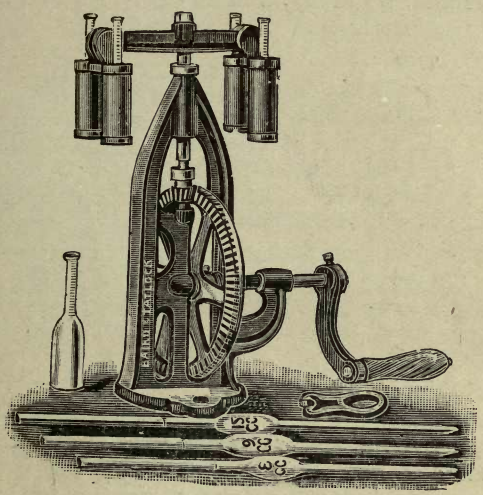


Fig. 13.—Leffmann and Beam Machine.

centrifuging, after dissolving everything in the milk but fat by strong sulphuric acid (the essential part of Babcock's method) with the addition of a little amyl alcohol to help the fat to separate (the essential part of Leffmann and Beam's method (Fig 13)). In chemical principles Gerber's method is nothing but Leffmann and Beam's, and a description of one only differs in details from that of the other; as, however, the details of Gerber's method render it more generally suitable, it will be described in preference to its predecessor.

Apparatus.—The essential apparatus consists of:

Acidobutyrometers or Test-bottles.—About 22 c.c. capacity with a long stem expanded to a conical bulb at the top, and graduated in percentages of fat; the bottom is open and contracted to a neck, which is closed by an india-rubber cork when in use. A suitable stand for these is provided.

The stems are made either round, flat, square, or with a magnifying scale; the patterns other than round give a wider and, by many, a more easily read scale.

The test-bottles can now be checked at the National

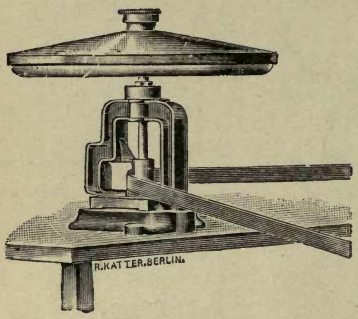


FIG. 14A.

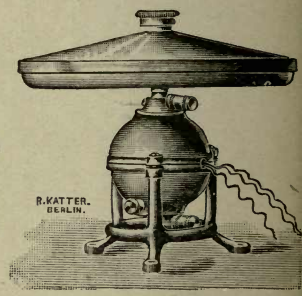


FIG. 14B.

Gerber Machine.

Physical Laboratory, who certify the correctness of the graduated scale.

Measuring Apparatus.—11 c.c. pipettes for measuring the volume of milk taken for the test; 10 c.c. pipettes for measuring the acid; and 1 c.c. pipettes for the amyl alcohol. Burettes or automatic measuring apparatus may be used.

Centrifuges.—A centrifuge is necessary to rapidly bring the fat into the graduated neck (Fig. 14A). In the smallest size this consists of two long cups hinged on a bar, which is fixed to the top of a vertical spindle running in plain bearings. This is driven by means of a strap round a loose pulley, or by means of a string. The larger machines have a disc carrying four or more cups, provided with a cover, and fixed to the top of a

vertical spindle running on ball bearings. The driving is performed either as in the two-bottle machine, by a string wound round the spindle or by a handle. Very large machines are fitted with a steam or water turbine, or an electro-motor (Fig. 14B), and may be provided with heating apparatus to keep the disc warm while running.

Hot-water Tank.—If the disc is not kept warm while running the bottles must be placed in water between 60° and 70° C., before reading, in a small tank.

The Process.—Place a sufficient number of bottles in the stand, open end upwards, and to each add 10 c.c. of sulphuric acid (*see* Appendix); add 11 c.c. of milk to each by means of the 11 c.c. pipette; then add 1 c.c. of amyl alcohol (*see* Appendix).

To measure with a pipette: place the constricted end in the liquid, and draw with the mouth till the level of the liquid is at least one inch above the mark on the upper stem; rapidly remove the mouth, and place the forefinger over the top; the finger must not be wet, though it may with advantage be slightly damp; if this is done with sufficient rapidity, the liquid is above the mark on the upper stem, if it is not it must be drawn up again. Carefully and slightly raise the finger to allow the liquid to run down slowly to the mark, then stop the flow by pressing the finger on the top; this operation requires some practice, but presents no real difficulty. Keeping the finger pressed down on the top, lift the pipette out of the liquid, and, taking care not to let any drops fall while moving, place the end of the pipette inside the neck of the bottle (Fig. 15); hold the pipette slanting, and let the point touch the side of the neck. On lifting up the forefinger the contents will run out down the side of the bottle. Let the pipette drain a few seconds, and remove it, but do not blow out the last drops. It is essential that the milk be measured with great accuracy, but there is less need for exactitude with the acid or amyl alcohol, as slight variations of these do not affect the results; it is as well, however, to cultivate a habit of accuracy.

After measuring the three liquids, which should float in three distinct layers, with little or no browning at the junction of the acid and milk, insert a cork; the bottle should be held with the left hand, and the

FIG. 15.—Measuring Milk.

cork screwed in, not pushed in, with the right; do not exert too much force or the bottle may break. If there is marked browning the milk has either been run in too fast or has not been run down the side of the bottle, and the experiment is best repeated.

Hold the bottle by the stem, and by the cork, keeping a slight pressure on the cork, and mix the contents by shaking; when all the white particles of curd have disappeared, but not before, invert the bottle to allow the acid to run out of the neck, and then turn it upright; repeat this several times till the acid that was in the neck has completely mixed. The bottle is now almost too hot to hold, from the heat developed by the action of the acid, and its colour is brown; place it in the centrifuge. Treat the other bottles in the same way, and arrange them in order in the centrifuge. It is well to mark each cup with a number to avoid errors.

The shaking of all the bottles may be done at once by means of the author's stand (Fig. 16), in which the bottles are held by being pushed into slits in the india-rubber plate; the hand should be placed over all the corks to prevent them from coming out, and the contents of the bottles from being spilt. Other convenient stands are also made.

If a number of samples insufficient to fill the disc is being tested, care should be taken to place them symmetrically so as to preserve the balance of the machine; a bottle filled with a mixture of equal parts of acid and water may be kept to make up an even number of bottles should an odd number of samples be tested.

Screw on the cover, and rotate the machine at about 1000 revolutions per minute for three or four minutes; modern centrifuges are usually turned with a handle; if there is a plain spindle, with a projecting piece, place the eye of the cord on the projection and turn the machine round counter-clock-wise till the cord is wound round the spindle; take the handle in the hand, pull hard, and the disc will spin; usually this must be done twice. If there is a strap, place this half round the pulley, and pull with the right hand in a somewhat downward direction, keeping the strap taut with the left hand, and continue giving sharp pulls till the disc spins rapidly; the pull must be downward as well as forward, or the pulley will not engage the spindle;

when the speed slackens, a few further pulls are necessary. If a string with two handles is provided, this is wound once completely round the spindle (or the pulley on the spindle); take a handle in each hand, and pull with the right hand, keeping the string taut with the left hand, and at the end of the pull continue the motion of the left hand to loosen the string round the spindle; pull back with the left hand keeping the string very loose, and then repeat the stroke. This method of driving requires care, but is, when learnt, a most satisfactory way of spinning the disc; when a sufficient speed is attained the string is allowed to hang loosely, the handles being placed on the bench at each side. If the string is not properly loosened at the end of the stroke, or on the return stroke, it winds up, and the handles must then be immediately dropped, or the hands may receive a nasty blow. Unbleached blind-cord is a suitable material for the string, and a good supply should be kept, as the string wears.

During the running the disc may be kept warm by means of a Bunsen burner or a spirit-lamp placed underneath near the edge, with a flame so adjusted that it just touches the disc.

The disc should be stopped gently, not suddenly, and the cover unscrewed. Do not take hold of the boss, in the centre of the cover, when the machine is running at high speed.

If the disc is not kept warm, place the bottles, after stopping the machine and removing the cover, in water kept at 60° to 70° C.; the small tank provided with the apparatus is nearly filled with water at the required temperature, and a spirit-lamp or Bunsen burner used to keep it warm.

After one minute the bottles may be read; hold the bottle (Fig. 16) by the top with the left hand and by the cork with the right, at a level with the eye; the position of the bottle should be as nearly vertical as possible. By gently moving the cork, the lower level

of the fat column is adjusted to one of the longer lines indicating percentages of fat, and the point on the scale where the lowest part of the curved upper limit stands

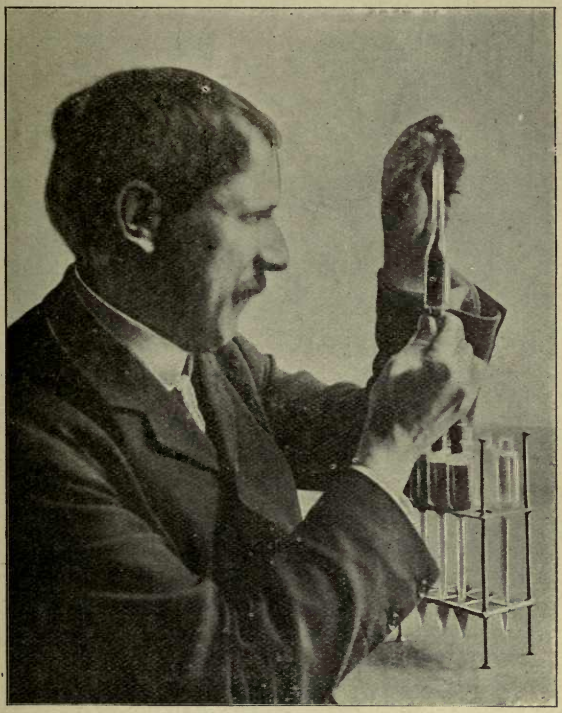


Fig. 16.—Reading Bottles.

is read off; a convenient method of reading is to count first the number of whole percentages of fat (indicated by the longer lines), and then the number of small lines above this, each small line indicating 0.1 per cent. of fat. Fig. 17 shows a sample reading 3.6 per cent. of fat; it is quite easy to read to half a small division or 0.05 per cent.

Every bottle should be read twice to make sure that there is no error in the first reading.

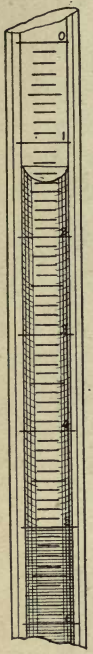


Fig. 17. Gerber Bottle.

The following are sources of error :

(*a*) Faulty graduation of the bottle ; this is rare.

(*b*) Chemicals not equal to specification ; it is important that the specification for these be strictly adhered to.

(*c*) Insufficient mixing of the milk, acid, and amyl alcohol ; this is indicated by the fat being cloudy, and obstinately refusing to become clear.

(*d*) Mixing of the milk and acid before addition of the amyl alcohol ; a brown colour of the fat is usually found here.

(*e*) Allowing a portion of the fat to remain in the little conical bulb ; if the fat is too low down on the scale for convenient reading, and the cork is not pushed in carefully, the fat is liable to jump up, as it is raised ; if the fat is too high, it may partially occupy the conical bulb ; in each case the fat must be allowed to run down before reading.

When the corks have been used for some time, they do not fit the necks of the bottles so well as when new, and are liable to slip out, and spill the acid mixture ; the times when they are most liable to come out are, when shaking the bottle, when removing it from the disc, when removing it from the warm water, and when pulling down the cork to adjust the fat layer. As acid is detrimental to the clothes, always keep a bottle of ammonia handy, and soak the acid-bespattered garment liberally with this, should any be spilt ; if acid is spilt on the hands or face, a copious stream of cold water is the remedy, but do not use ammonia ; even strong sulphuric acid spilt on the flesh rarely does harm if washed off at once, and plenty of water is used.

When the bottles have been read, turn them cork upwards in the stand, remove the corks, and empty the contents of the bottle into a convenient pot, not down a sink. The pot when full may be emptied down a well-flushed drain; rinse the bottles two or three times in hot water and leave to drain open end downwards; an occasional clean with a brush when the inside becomes dingy is necessary. The corks should be put in a basin and washed several times with hot water and allowed to dry.

Care of the Machine.—The bearings require lubrication; an oil-can and a supply of suitable oil is provided with the machine. If the ball-bearings wear loose, they can be adjusted by means of a collar in the top bearing. If a bottle breaks in the machine remove the cup and wash it carefully, and should acid be spilt on the disc, wash this too.

The machine should be clamped and screwed to a firm bench or table, preferably close to a leg.

If separated milk is being examined a special bottle with narrow neck should be used, and the time of centrifuging increased.

Cream cannot be tested direct, as the scale does not go beyond 9 per cent., or in some bottles 6 or 7 per cent., and a special bottle may be used. But cream can be tested in the ordinary bottles by the following method; if a thin cream (containing less than 32 per cent. fat) is to be examined, use a 3 c.c. pipette in place of the 11 c.c. pipette; fill the bottle with acid as before, and then add 8.2 c.c. of water from a pipette graduated in $\frac{1}{10}$ c.c.; fill the 3 c.c. pipette with cream, adjusting the upper level very accurately to the mark, and wipe the outside of the stem; hold the pipette vertically over the centre of the opening and allow the cream to run directly into the water and blow out the last drops; finally, add the amyl alcohol, and proceed as described for milk.

For thicker creams, a small balance weighing to 0.05 gramme (Fig. 18), and a pair or pairs of accurately

balanced tin pots are necessary; place sufficient cream in one pot to fill it nearly half full, and put this on one pan of the balance; place the other pot on the other pan and pour in separated milk or

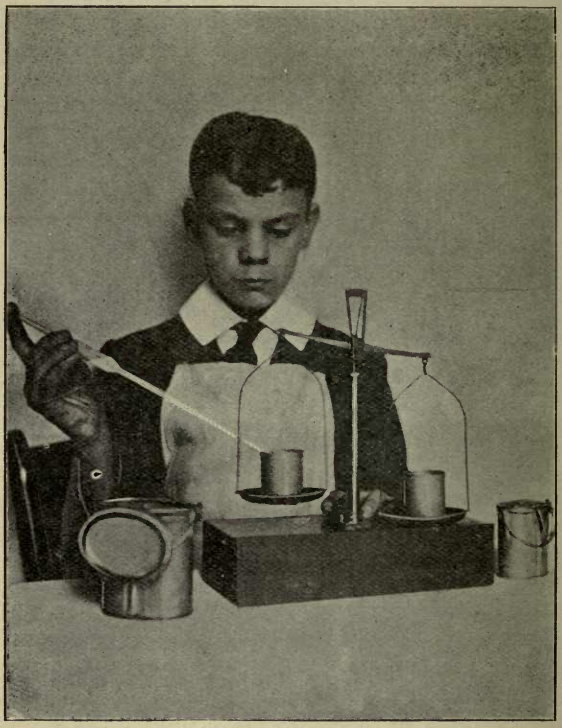


Fig. 18.—Weighing Cream.

water till exactly balanced. Mix the contents of the two pots by pouring backwards and forwards several times, and measure out the mixture as directed for thin cream.

Use the following Table for the calculation of results; the first column is the reading observed; if thin

cream was tested, its percentage of fat is found in the column headed "Undiluted"; if thick cream was examined, the column headed "Diluted" will give the percentage of fat.

TABLE III

FOR CALCULATING FAT IN CREAM

The Table should be checked by a gravimetric method, and may require a slight correction added or subtracted which may vary with each pipette.

Reading	Diluted	Undiluted	Reading	Diluted	Undiluted
8.5	63.5	31.8	6.7	49.4	24.8
8.4	62.7	31.4	6.6	48.7	24.4
8.3	61.9	31.0	6.5	47.9	24.0
8.2	61.1	30.6	6.4	47.2	23.7
8.1	60.3	30.2	6.3	46.4	23.3
8.0	59.5	29.8	6.2	45.7	22.9
7.9	58.8	29.5	6.1	44.9	22.5
7.8	58.0	29.1	6.0	44.1	22.1
7.7	57.2	28.7	5.9	43.4	21.8
7.6	56.4	28.3	5.8	42.6	21.4
7.5	55.6	27.9	5.7	41.8	21.0
7.4	54.8	27.5	5.6	41.1	20.6
7.3	54.0	27.1	5.5	40.3	20.2
7.2	53.3	26.7	5.4	39.5	19.8
7.1	52.5	26.3	5.3	38.8	19.5
7.0	51.7	25.9	5.2	38.0	19.1
6.9	50.9	25.5	5.1	37.2	18.7
6.8	50.2	25.2	5.0	36.4	18.3

In the "Sinacid," "Sal," and "Neusal" methods an alkaline solution is substituted for the acid of the Gerber process, and iso-butyl alcohol for the amyl alcohol. Gerber bottles are used for these methods; 11 c.c. of alkaline solution, 10 c.c. of milk, and 0.6 c.c. of iso-butyl alcohol (which is usually coloured by a dye) are measured into the tubes. The mixing and centrifuging are done as in the Gerber method, but the temperature of the water-bath is 45° C. The coloured alcohol passes into the fat, and colours the layer, which is consequently easy to read.

While the substitution of the alkaline solution avoids the objections to a strong acid, this method has the drawback that the corks become slippery and tend to come out of the bottles.

Gravimetric Estimation of Fat.—Of the numerous methods for the estimation of fat, the Storch, the Ritthausen, the Werner-Schmid, and the Gottlieb methods all present advantages.

The Storch Method.—Place three or four grammes of ignited kieselguhr in a basin, and pipette 10 c.c. of milk in such a manner that it is all absorbed by the kieselguhr; dry on the water-bath with occasional stirring to break up lumps, grind fine, and transfer to a fat-free thimble, rinsing out the basin with fresh quantities of kieselguhr; place the thimble in a Soxhlet extractor, rinse the basin, pestle, &c., with ether, and pour this into the thimble; extract the kieselguhr with ether.

The Ritthausen Method.—*See* Estimation of Proteins, p. 34.

The extraction in these methods is performed by attaching a weighed flask to the bottom of the extractor to receive the ether containing the fat, and connecting the extractor to an upright condenser; the flask is immersed in water kept warm by a small flame, and the ether continually distils up, is condensed, and runs back into the extractor, from which it siphons back into the flask when the extractor is full.

The kieselguhr should be extracted about four hours, and casein about one and a half hours, after which the ether is distilled from the flask, and the fat freed from ether by placing in a water-oven for twenty minutes, blowing into and rotating the flask every five minutes. After cooling for a quarter of an hour, the flask is weighed, and the increase of weight represents the fat.

It is advisable to return the flask to the water-oven for a second period, and re-weigh to make sure that all the solvent has been removed. The fat may be melted and dissolved in petroleum ether which is

carefully decanted from any residue; by successive treatments with small quantities of petroleum ether all the fat may be removed, and the flask, with any

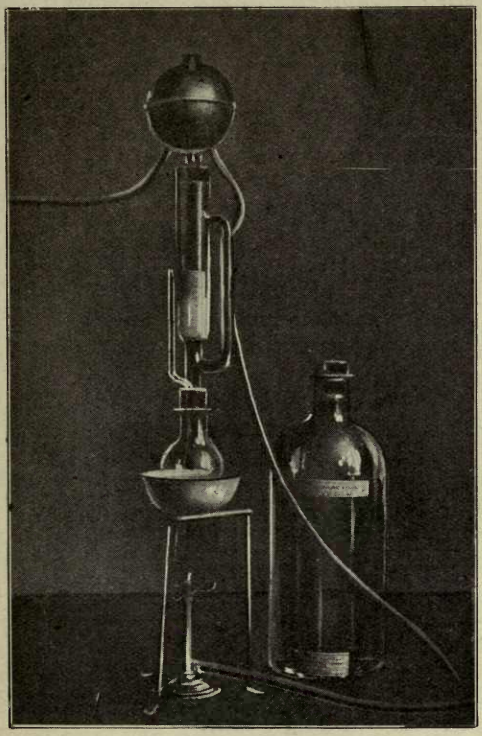


Fig. 19.—Soxhlet Extractor.

small particles of kieselguhr that may have passed into it, re-weighed.

The Werner-Schmid Method.—Pipette 10 c.c. of milk into a Stokes tube (Fig. 20), add 10 c.c. of strong hydrochloric acid, and heat over a flame *with constant shaking*, till the fat, on standing a short time, collects in a clear layer on the surface; cool the contents of the

tube, and add 30 c.c. of ether, cork the tube, and shake well; allow the tube to stand till the ether has separated in a clear layer; remove as much ether as possible, preferably by means of washbottle tubes, to a weighed flask; add about 20 c.c. more ether, shake well, allow the ether to settle clear, and remove as before, and again add about 20 c.c. of ether, shake, allow to settle, and remove the clear layer. Distil off the ether, dry, and weigh as above.

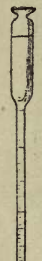


FIG. 20. Stokes Tube

Gottlieb's Method.—Place 5 c.c. of milk in a stoppered tube, add successively 0.5 c.c. of ammonia, 5 c.c. of alcohol, 12½ c.c. of ether, and 12½ c.c. of petroleum ether; mix after each addition, and allow to stand; when a clear layer has separated, mix again, allow to separate, mix once more, and allow the clear layer to completely separate.

Remove the ethereal layer as completely as possible by washbottle tubes to a flask, and add and remove three successive portions of a mixture of equal parts of ether and petroleum ether. Evaporate the solvent, dry, and weigh as above. Extract with petroleum ether and weigh the empty flask.

Estimation of Milk Sugar.—This may be estimated either polarimetrically, or gravimetrically; the results in either case are expressed as anhydrous sugar.

Polarimetric Estimation.—50 c.c. of milk are measured into a dry flask, and a quantity of water equal in cubic centimetres to the sum of

(*a*) The degrees of gravity divided by 20.

(*b*) The percentage of fat divided by 1.8.

(*c*) A quantity to convert scale readings into percentages of anhydrous sugar; if the scale is in angular degrees and a 200 mm. tube is used, this is 5.43 c.c. (or 5 c.c. with a 198.4 mm. tube).

1.5 c.c. of Wiley's acid mercuric nitrate solution (*see* Appendix) is added, and the whole well mixed by

violent shaking. The solution is poured on a dry filter, and a polarimeter tube filled with the clear filtrate.

As an example : the milk has a sp. gr. of 1.032, the degrees of gravity are 32.0, and (*a*) is $\frac{32.0}{20} = 1.60$ c.c.; the fat is 3.60 and (*b*) is $\frac{3.6}{1.8} = 2.00$ c.c.; if an instrument graduated in angular degrees and a 200 mm. tube are used (*c*) is 5.43. The water added is 1.60+2.00+5.43= 9.03 c.c.

The reading is made by placing the tube in the instrument (Fig. 21), focusing the eye-piece on the half-shadow plate when the analyser is so turned that one side is darker than the other, and adjusting the analyser till both sides are equal in intensity ; the scale is then read by means of the vernier provided. Several readings should be made, the adjustment to equality being made from either side alternately, and the mean of the readings taken as the correct reading. A blank estimation, *i.e.* one with a tube filled with distilled water, should always be made, and the reading if to the right subtracted from, or if to the left added to, the reading of the sample. The corrected reading of the scale gives the percentage by weight of anhydrous milk sugar.

Mercuric nitrate does not remove the proteins quite completely ; for greater accuracy add to the filtrate $\frac{1}{20}$ of its volume each of phospho-tungstic acid solution and of dilute (1 : 1) sulphuric acid solution, filter and polarise. Multiply the reading by 1.1. The error with whole milk is, however, very small and hardly exceeds the experimental error of reading.

Gravimetric Estimation.—About ten grammes of milk are placed in a 100 c.c. flask with 60 to 70 c.c. water, 5 c.c. of Fehling's copper sulphate solution added (*see* Appendix), and the solution neutralised with caustic soda ; the liquid is made up to 100 c.c., and the contents of the flask, after mixing, filtered through a dry filter ;

50 c.c. of the filtrate are placed in a beaker, and a mixture of 30 c.c. each of Fehling's copper sulphate and alkaline tartrate solutions (*see* Appendix) added; the beaker is heated by a flame of such size that the liquid

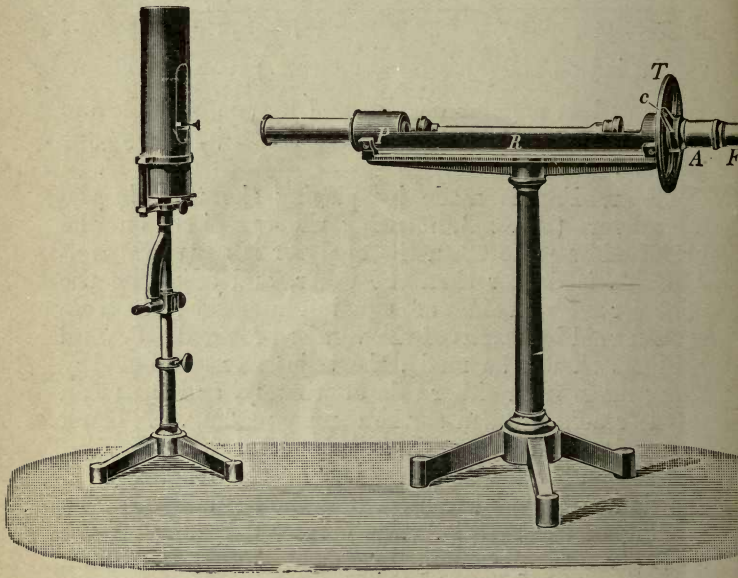


FIG. 21.—Polariscope.

boils in about four minutes, and it is kept boiling for exactly six minutes. A tube of hard glass, about 1 c.m. in internal diameter and 10 c.m. long, with one end drawn out, and plugged with a fairly tight wad of asbestos, is ignited, cooled, and weighed; the drawn-out end is inserted in a hole in the cork of a vessel from which the air can be exhausted (*e.g.* by a filter pump), a small funnel is placed in the wider end, and the liquid carefully poured off from the red precipitate of cuprous oxide; the precipitate is washed several times by decantation with hot, well-boiled water, and is then washed into the tube, and the last traces removed

from the sides of the beaker by rubbing with a policeman, and transferred to the tube; the precipitate in the tube is well washed with hot water, and finally with alcohol, and dried. The narrow end of the tube is

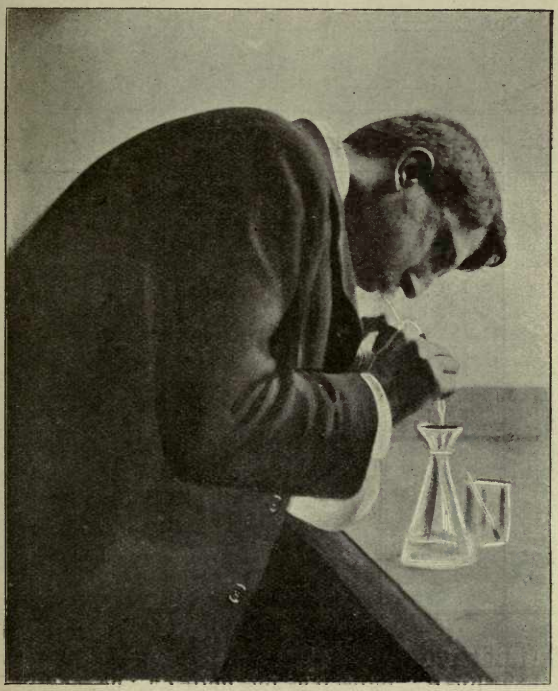


Fig. 22.—Washing Precipitate.

connected with an apparatus from which hydrogen can be evolved, and after the stream has passed for a few minutes the part of the tube containing the precipitate is gently heated by a small flame, till the cuprous oxide is reduced to copper; the hydrogen is passed till the tube is cool, and it is then disconnected and weighed; the increase in weight gives the quantity of copper,

from which the quantity of milk sugar can be found by the table below; this multiplied by 200, and divided by the weight of milk taken, gives the percentage of milk sugar.

TABLE IV

FOR CALCULATING WEIGHT OF MILK SUGAR FROM COPPER REDUCED

(All weights are in milligrammes)

Copper	Milk Sugar	Copper	Milk Sugar	Copper	Milk Sugar
120	86.4	215	158.2	310	232.2
125	90.1	220	161.9	315	236.1
130	93.8	225	165.7	320	240.0
135	97.6	230	169.4	325	243.9
140	101.3	235	173.1	330	247.7
145	105.1	240	176.9	335	251.6
150	108.8	245	180.8	340	255.7
155	112.6	250	184.8	345	259.8
160	116.4	255	188.7	350	263.9
165	120.2	260	192.5	355	268.0
170	123.9	265	196.4	360	272.1
175	127.8	270	200.3	365	276.2
180	131.6	275	204.3	370	280.5
185	135.4	280	208.3	375	284.8
190	139.3	285	212.3	380	289.1
195	143.1	290	216.3	385	293.4
200	146.9	295	220.3	390	297.7
205	150.7	300	224.4	395	302.0
210	154.5	305	228.3	400	306.3

Estimation of Proteins.—These may be estimated together by Ritthausen's method, or the casein and albumin may be separated; indirect estimations may be made from the nitrogen by Kjeldahl's method, from the "aldehyde figure," or from the organic phosphorus and sulphur.

The Ritthausen Method.—Pipette 10 c.c. of milk into a beaker, and add 100 c.c. of hot water; add 5 c.c. of Fehling's copper sulphate solution (*see* Appendix), and neutralise with caustic soda solution; collect the precipitate either in a weighed Gooch crucible, or on

tared filter-paper; remove the precipitate completely from the beaker by means of a policeman, and wash well (Fig. 22). Dry in the water-oven, and extract the fat with ether, preferably in a Soxhlet extractor, and dry again till the weight is constant. Ignite the precipitate in the Gooch crucible, or place the filter containing the precipitate in a weighed basin and ignite it (if tared filters are used the tare should also be ignited in a weighed basin); subtract the weight of the ash (corrected if necessary for the ash of the tare) from the weight of the precipitate, and the difference will give the weight of the proteins.

Estimation of Casein and Albumin.—Pipette 10 c.c. of milk into a beaker, add 90 c.c. of water at 42°–43° C. and 1.5 c.c. of a 10 per cent. solution of acetic acid; stir well, and collect and weigh the precipitate as above. [*Note.*—This estimation or that given above may be combined with a fat estimation.] The precipitate is in this case casein.

Raise the filtrate to boiling, and keep for 15 minutes on the water-bath; collect the precipitate as above, and weigh after drying; as fat and ash are absent the extraction and ignition may be omitted. The precipitate consists of albumin.

Estimation of Nitrogen by Kjeldahl's Method.—5 c.c., or 10 c.c., of milk are pipetted into a long-necked hard glass flask; 20 c.c. pure sulphuric acid and a drop of mercury are added; a long-stemmed bulb is placed in the neck; the flask is supported in an inclined position, and it is heated by a small flame; at first water is driven off, next a considerable amount of frothing takes place, and when this has subsided the flame may be turned up to such a height that the sulphuric acid distils up to and is condensed in the neck; 10 grammes of acid potassium sulphate may be added after the frothing has subsided, but this addition is hardly worth while with milk, as the operation is fast enough without it; the heating is continued till the liquid in the flask is quite colourless.

After cooling, the acid liquid is diluted with about 100 c.c. of water, and poured into a flask of at least 1000 c.c. capacity (preferably of copper (Fig. 23)), and

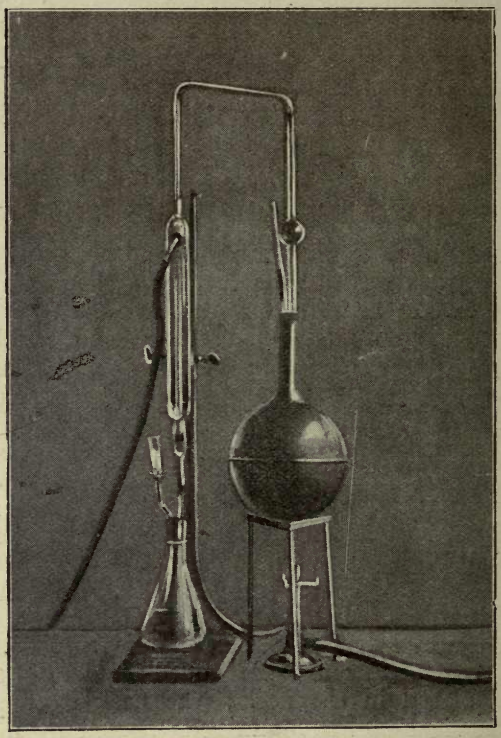


Fig. 23.—Kjeldahl Apparatus.

the hard glass flask rinsed out with further quantities of water; the large flask is furnished with a cork carrying a tube which is connected to a condenser, and a tube allowing additions to be made to the liquid. 25 c.c., (50 c.c. if 10 c.c. of milk was taken), of $\frac{N}{10}$ acid, or an amount calculated to be slightly more than sufficient from

the aldehyde figure (p. 38), are accurately measured into a flask, which is placed so that the end of the condenser dips into the liquid. Through the tube in the cork 100 c.c. of a solution of caustic soda containing 300 grammes per litre are poured in, followed by 10 c.c. of a 10 per cent. solution of sodium sulphide ; the tube is closed, and the contents of the flask mixed by shaking ; a flame is placed under the flask, and the ammonia formed by the action of the sulphuric acid on the proteins and liberated by the alkali, distilled over ; the distillation takes about half an hour, a volume of approximately 200 c.c. being collected. A few drops of cochineal are added to the distillate, and the excess of acid titrated with $\frac{N}{10}$ alkali (*see* Appendix). A blank experiment, *i.e.* one without the milk, should be performed, and the difference between the volume of alkali used in the blank and the experiment in cubic centimetres multiplied by 0.0014 will give the weight of nitrogen ; this multiplied by 100 and divided by the weight of the 5 c.c. taken will give the percentage of nitrogen ; the nitrogen multiplied by 6.38 may be taken as proteins in the milk.

Estimation of Casein and Albumin Nitrogen.—To 10 c.c. of milk add 20 c.c. of a saturated solution of magnesium sulphate, and crystals of the salt so long as they are dissolved ; allow to stand for some time, and filter off the precipitated casein, and wash this several times with the saturated solution of magnesium sulphate (this is a slow process) ; place the filter and precipitate in a long-necked, hard glass flask, add 30 c.c. of pure sulphuric acid and a drop of mercury, and proceed as above ; 150 c.c. of soda solution must be added before distillation, and the distillate should be collected in 35 or 40 c.c. of $\frac{N}{10}$ acid.

The filtrate is diluted, and the albumin precipitated by tannin ; the precipitate is collected on a filter, and the nitrogen estimated as above.

The casein nitrogen and albumin nitrogen may be multiplied by 6.38 to give the amounts of casein and albumin respectively; the sum of the two is usually a little below the total nitrogen.

Indirect Estimation from Organic Phosphorus and Sulphur.—Estimate the phosphoric acid in the ash of the milk (*see* p. 15), preferably using 25 grammes of milk; evaporate 25 c.c. of the filtrate obtained by adding Wiley's mercuric nitrate to milk for the polarimetric estimation of milk sugar (*see* p. 30), ignite and estimate the phosphoric acid in the ash of this; multiply the weight of P_2O_5 by 4.42, and this will give the percentage of mineral phosphoric acid in the milk; this subtracted from the percentages of total phosphoric acid will give the organic phosphoric acid, and the casein can be calculated from this by multiplying by 50.8.

Take 25 c.c. of milk, add 10 c.c. of strong nitric acid and 1 gramme of sodium carbonate, evaporate to dryness, and add 2 or 3 c.c. more nitric acid, and again evaporate; ignite to a fairly white ash, and take up the residue with dilute hydrochloric acid; evaporate to dryness, and boil the residue again with dilute hydrochloric acid, and filter. Just raise the filtrate to boiling, and add barium chloride solution drop by drop so long as a precipitate is produced. Allow the solution to stand 24 hours to complete the precipitation, the first two hours preferably in the water-oven, and collect the precipitate on a small filter, wash well with hot water, and place filter and precipitate still wet in a weighed platinum dish; ignite over a small flame till the paper is burnt, and then more strongly; the weight of the precipitate (corrected for the ash of the filter) multiplied by 13.7 and divided by the weight of milk taken will give the percentage of sulphur in the milk. For each 71 parts of organic P_2O_5, 32 parts of sulphur belong to the casein; the remainder of the sulphur multiplied by 58.5 will give the albumin.

Aldehyde Figure.—This determination may be combined with an acidity estimation; to 10 or 11 c.c.

of milk at least 1 c.c. of 0.5 per cent. phenolphthalein solution is added, and the milk neutralised with $\frac{N}{11}$ (approximately) strontia solution; to the faintly pink liquid 2 c.c. or more of Schering's 40 per cent. formaldehyde solution is added, and the titration continued till the same degree of pink colour appears. After deducting the acidity of the formaldehyde solution, the latter titration represents the aldehyde figure. The aldehyde figure is obtained by multiplying the number of c.c. used by the strength of the solution (*see* p. 95) and by 1000, and dividing by the volume taken. Proteins may be deduced from this figure by multiplying by 0.170; this factor is only applicable to fresh cow's milk when determined by strontia solution.

Catalase.—For the estimation, the measuring cylinder is filled with water through the opening *d*, the cover of which is then screwed down. The opening *b* (for cleaning the tube) is closed with a rubber cork, and the chamber *A* is charged, through the opening *c*, with 15 c.c. of the milk and 5 c.c. of 1 per cent. hydrogen peroxide solution (or 9 c.c. of milk and 3 c.c. of hydrogen peroxide). The tube is now held at *f* and *d* and shaken with a pendulum motion, and the cover to *c* rapidly screwed down. The tube is then placed in water at 25° C. up to the level of *c*, shaken from time to time, and the volume of the liberated gas which rises into the measuring chamber *B* read off after two hours.

FIG. 24.—Lobeck's Catalase Tube.

The Relation between Fat, Solids not Fat, and Specific Gravity.—As the solids not fat of milk are heavier than water, and the fat is lighter, and as, moreover, the extent to which each of these is heavier or lighter respectively

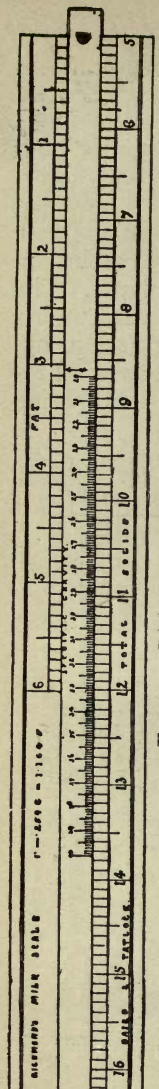


FIG. 25.—Richmond's Milk Scale.

is practically constant, it is evident that by means of an approximate formula any one of the three can be approximately calculated from the other two.

A simple formula which gives a very good approximation is :

$$S = \frac{G}{4} + \frac{F}{5} + 0.14 \quad . \quad . \quad (1)$$

where S = Solids not Fat per cent. by weight ;

G = degrees of gravity ; and

F = Fat per cent. by weight.

If it is desired to calculate directly the Total Solids (T), which consists of the sum of the fat and solids not fat, we may write

$$T = S + F = \frac{G}{4} + 1.2\,F + 0.14 \quad . \quad (2)$$

As the solids not fat are not generally estimated directly, but are obtained by subtracting the fat from the total solids, the second formula is more useful when the fat is to be calculated from the specific gravity and total solids, and it may be usefully converted to the form

$$F = 0.833\,T - 0.208\,G - 0.12 \quad . \quad (3)$$

or perhaps more simply

$$1.2\,F = T - \frac{G}{4} - 0.14 \quad . \quad . \quad (4)$$

Tables for the easy calculation of solids not fat from fat and specific gravity, and fat from total solids and specific gravity are given at the end of the book (pp. 89, 90). As the method is only approximate the figures are calculated only to the nearest 0.05 per cent. To use these Tables, find in the upmost

horizontal column the specific gravity, and in the vertical column the fat or the total solids; in the space where the columns intersect the figure required is found. If the last figure in the specific gravity is 9, 0, or 1, or 4, 5, or 6, use the figure in the column corresponding

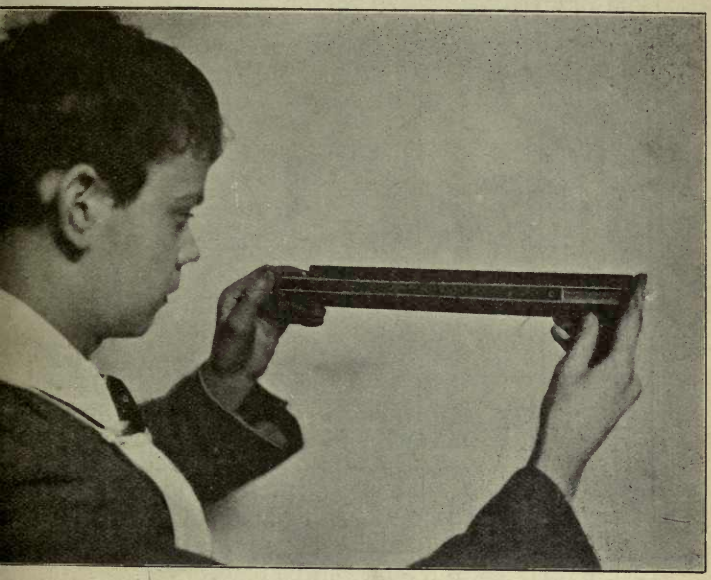


Fig. 26.—Using Milk Scale.

to the nearest 0 or 5; if it is 2 or 7, use the column corresponding to 0 or 5, but add in Table XII 0.05, and subtract 0.05 in Table XIII; if it is 3 or 8, use the column corresponding to 5 or 0, and subtract 0.05 in Table XII and add 0.05 in Table XIII. Read directions for use of each Table.

The calculation may also be made with the milk scale, which consists of a slide rule (Figs. 25, 26); on one side is marked total solids (1 inch = 1 per cent.), on the other the fat (1.2 inches = 1 per cent.), on the slide

is marked specific gravity ($\frac{1}{4}$ inch = 1 degree), and an arrow is placed 0.14 inch from the end of the scale. If the arrow is placed against the fat, the specific gravity lies against the total solids; *vice versâ*, if the specific gravity is placed against the total solids the arrow will point to the fat.

On another part of the rule is a scale for correcting the specific gravity taken at any temperature to 60° F. This is not shown in Fig. 25. On the rule is a scale of degrees of gravity (marked lactometer degrees), and

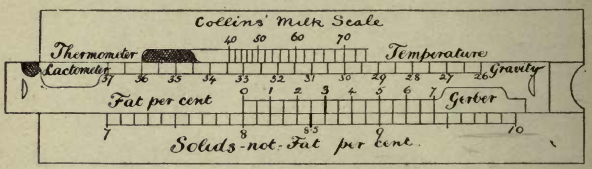


FIG. 27.—Collins' Milk Scale

on the slide a scale of temperature; an arrow is placed at 60° F. and this is placed against the degrees of gravity found; the temperature then corresponds to the specific gravity corrected to 60° F.; the readings from the milk scale only deviate from those given by the Table within the limits of reading a lactometer.

Collins has devised a milk scale (Fig. 27) by which the percentage of solids not fat can be read direct. In this it is only necessary to set the reading on the lactometer opposite the temperature at which the specific gravity was taken, when the percentage of solids not fat will be found opposite the percentage of fat.

Though the calculation of the solids not fat or fat can never be so exact as the direct estimation, it is sufficient for many purposes, where small deviations do not affect the conclusions drawn from the results; this method also provides a useful check on the analyses where all three estimations are made, and it is hardly ever found that the deviations of the calculated figures from those estimated exceed 0.2 per cent.

CHAPTER III

THE ANALYSIS OF MILK-PRODUCTS

The liquid milk-products are skim-milk, cream, whey, butter-milk, sterilised milk, condensed milk, and sour or fermented milk; milk powder consists of milk evaporated to dryness.

Skim-milk is treated exactly as milk; as the fat globules are very small and few, the estimation of fat requires more care; the period of revolution of the disc in the Gerber method should be increased, and a correction of 0.05 per cent. should be added to the reading unless a special tube is used. The gravimetric methods of Storch and Ritthausen tend to give slightly low results, and those of Werner-Schmid or Gottlieb are preferable. In the estimation of the proteins by Ritthausen's method, the extraction of the fat may be omitted, and the percentage of fat found subtracted from the results.

Cream requires several modifications. The *specific gravity*, except of a thin cream, is difficult to estimate, and this is usually omitted. It is advantageous to add an equal volume of alcohol to the cream before drying for the *total solid estimation*, as there is then no skin to be broken. The *fat* may be estimated by macerating the total solids with ether or amyl alcohol, carefully decanting and repeating the maceration, &c., about half a dozen times; the solids not fat are weighed directly, and the fat found by difference. The *ash estimation* is made on the solids not fat. The Werner-Schmid method is, however, excellent for cream, with the modification that 2 or 3 grammes should be weighed, into the Stokes tube, and the weight made up to 10 grammes with water. The Gottlieb method is also good; 1 to 2 grammes should be weighed, and enough water added

to make the total weight up to 5.15 grammes. The methods are then carried out as for milk.

The fat may be calculated from the total solids with a close approach to accuracy by the formula

$$F = 1.104\ T - 10.4$$

or by the following Table :

TABLE V

FOR CALCULATING PERCENTAGE OF FAT IN CREAM FROM TOTAL SOLIDS

Total Solids	Fat	Solids not Fat	Total Solids	Fat	Solids not Fat
60	55.8	4.2	44	38.2	5.8
59	54.7	4.3	43	37.1	5.9
58	53.6	4.4	42	36.0	6.0
57	52.5	4.5	41	34.9	6.1
56	51.4	4.6	40	33.8	6.2
55	50.3	4.7	39	32.7	6.3
54	49.2	4.8	38	31.6	6.4
53	48.1	4.9	37	30.4	6.6
52	47.0	5.0	36	29.3	6.7
51	45.9	5.1	35	28.2	6.8
50	44.8	5.2	34	27.1	6.9
49	43.7	5.3	33	26.0	7.0
48	42.6	5.4	32	24.9	7.1
47	41.5	5.5	31	23.8	7.2
46	40.4	5.6	30	22.7	7.3
45	39.3	5.7	29	21.6	7.4

This method is not available in the case of clotted or Devonshire cream.

The aldehyde figure multiplied by 0.45 will approximate to the solids not fat in both fresh and clotted creams.

The **milk-sugar estimation** by the polariscope requires modification, as the cream must be more highly diluted ; it is best to weigh out 50 grammes of cream, make up to 100 c.c., and add 1 c.c. of Wiley's acid mercuric nitrate, and polarise.

The milk sugar is found by the formula

$$\text{Milk sugar} = R \times \frac{1}{1.05} \times 0.95 \times \frac{100 - 1.076\,F \times W}{W}$$

Where R = Reading in angular degrees with a 200 mm. tube.
F = percentage of fat by weight.
W = weight of cream taken.

The gravimetric estimation will require slight correction for the volume of the fat, and the formula

$$\text{Milk sugar} = 2M \times \frac{100 - 1.076\,F \times W}{W}$$ may be used.

M = weight of milk sugar obtained from the Table on p. 34.

The **protein estimation** is carried out as for milk; it saves time, however, to dry the cream and to extract the bulk of the fat before submitting the sample to Kjeldahl's method, as fat is attacked but slowly by sulphuric acid and mercury.

Butter-milk is analysed in exactly the same way as milk.

It sometimes happens that when churning, both salt and water find their way into the butter-milk; when the butter-milk is to be sold, it is important to be able to rapidly estimate both the proportion of water and of salt.

Chlorides can be titrated in milk with $\frac{N}{10}$ silver nitrate solution, using potassium chromate as indicator, but as milk itself contains chlorides a correction is necessary for this. To 10 c.c. add a few drops of potassium chromate solution and run in $\frac{N}{10}$ silver nitrate (*see* Appendix) till a faint red tinge is seen; from the quantity used subtract the aldehyde figure (obtained with strontia) multiplied by 0.171; the remainder multiplied by 0.0585 gives the sodium chloride.

By multiplying the amount of salt found by 0.00735

the increment of specific gravity due to the addition is deduced, and subtracting this from the specific gravity found, the specific gravity of the milk is obtained. From this last figure and the fat the solids not fat can be calculated, and from this the amount of added water deduced by the formula on p. 52, or the formula which is based on the above factors,

$$\text{added water} = \frac{36 - \left(\text{G} + \text{F} - \frac{\text{c.c. } \frac{\text{N}}{10} \text{ silver solution} - \text{aldehyde fig.} \times 0.171}{2.3}\right)}{36} \times 100$$

may be used.

Whey is treated as milk; it contains, however, no casein, but gives a small precipitate consisting of albumoses, which by the methods given would be estimated as casein. The aldehyde figure multiplied by 0.125 gives the percentage of proteins.

Sterilised milk can be analysed by the methods given for milk; the polarimetric estimation of milk sugar tends to be low, owing to change in the milk sugar on heating, and the gravimetric method should be used. The albumin behaves like casein, as it is rendered insoluble in dilute acetic acid and magnesium sulphate solutions; the estimation of casein and albumin can, however, be made by the indirect method from organic phosphorus and sulphur. The total nitrogen is unaffected. Ritthausen's method should not be used for the estimation of fat in sterilised or condensed milk.

Condensed milk, if unsweetened, may be analysed by diluting one part by weight with two parts of water, and boiling and treating in the same way as sterilised milk; the results must, of course, be multiplied by 3.

Sweetened condensed milk should be similarly diluted and analysed. Great care must be taken to well mix the contents of the tin, as the milk sugar

separates in minute crystals, which on long standing sink to the bottom. It is not generally heated to an extent sufficient to affect the milk sugar, and milk and cane sugar may be estimated by Harrison's method.

Harrison's Method.—Prepare the filtrate with acid mercuric nitrate as usual (p. 30), taking, however, double quantities; place as much of the filtrate as possible in a 100 c.c. flask and weigh this; immerse for exactly seven minutes in a briskly boiling water-bath to invert the cane sugar, cool, and make up with distilled water to the original weight. Fill the 200 c.c. tube with this solution (cleared if necessary by filtration), polarise and at once note the temperature. Multiply the difference between the direct and the inverted readings by $\frac{100}{142.66 - \frac{t}{2}}$, and this will give the reading due to cane sugar; this divided by 1.2 will give the percentage of cane sugar in the diluted milk; the percentage of anhydrous milk sugar in the diluted milk is given by the difference between the direct reading and that due to cane sugar. These percentages multiplied by 3 will give the amounts in the condensed milk.

The Werner-Schmid method for the estimation of fat should not be used, and the Gottlieb method is recommended.

Sour milk is difficult to analyse, and the results are generally less satisfactory than those obtained with fresh milk. If approximate results only are wanted, such as would be furnished by a determination of specific gravity and fat alone, the following modification of Weibull's method may be used; measure the sour milk, and to each 100 c.c. add 5 c.c. of a solution of ammonia (1 part of ammonia, sp. gr. 0.880, to 4 parts water); shake gently, and allow to stand till the precipitated casein is all dissolved; the specific gravity is estimated by a lactometer, and a correction (usually about 2.7 or 2.8 degrees), found experimentally by noting the decrease of specific gravity in fresh milk

treated similarly, is added; the fat is estimated by the Gerber method (or one of the gravimetric methods), and the result is increased by one-twentieth; this method is available if the milk is not too old, and serves excellently for control work.

An estimation of total solids may be made after neutralising the acid with strontia solution; an estimation of the acidity is made as usual (p. 15), a weighed quantity being used instead of a measured amount, and a proportionate amount of strontia solution added to the weighed quantity of milk taken for total solid estimation; from the weight of total solids 0.00428 gramme should be deducted for each cubic centimetre of $\frac{N}{10}$ strontia added.

The ash is estimated as usual, but 0.00738 gramme should be deducted from the weight of the ash for each cubic centimetre of $\frac{N}{10}$ strontia solution added.

The fat estimation is preferably made by the Storch method (an addition of strontia solution being made as in the total solid estimation) or the Gottlieb method; the Werner-Schmid method is also available, though it tends to give high results with very sour samples owing to the solubility of lactic acid in ether.

Other determinations are made as for milk, except that the quantities taken are all weighed and not measured. The total nitrogen is a useful datum.

The aldehyde method does not give exact results for proteins.

The methods used in the Government laboratory include the determination of alcohol, volatile acids, and ammonia, and from these, the solids lost by the various fermentations undergone by the milk are reconstructed. The following description is condensed from Dr. Thorpe's report:

Alcohol.—To 75 grammes of sour milk half the caustic soda solution necessary to neutralise is added, and the mixture is distilled; to the distillate is added

0.5 c.c. $\frac{N}{10}$ soda solution, and this mixture again distilled; the final distillate is made up to the original bulk, and the specific gravity estimated. The difference in degrees of gravity between the specific gravity and 1000 multiplied by 0.977 gives the percentage by weight of milk sugar converted into alcohol.

Volatile Acid. — Ten grammes of milk are neutralised to the extent of one-half, and a little phenolphthalein added; the mixture is evaporated to dryness on a water-bath with frequent stirring, and after the addition of 20 c.c. boiling distilled water, $\frac{N}{10}$ soda solution is added till a pink colour just appears. The difference between the number of c.c. of $\frac{N}{10}$ soda solution used in this experiment, and that required for the original acidity of 10 grammes of milk, is multiplied by 0.0255 to give the percentage of milk sugar converted into volatile acid.

Ammonia.—Two grammes of milk are diluted to 100 c.c. and filtered clear. Ten c.c. of the filtrate made up to 50 c.c. with distilled water are compared in tint with a solution of ammonium chloride solution (1 c.c. = 0.01 milligramme (NH_3) in 50 c.c. water containing 10 c.c. of a solution of 2 grammes fresh milk acidified in 100 c.c. after the addition of 2 c.c. of Nessler solution (*see* Appendix) to each. The number of c.c. of ammonia solution required to produce the same tint multiplied by 0.026 gives the percentage of casein converted into ammonia.

The three amounts are added together, and constitute the total correction for solids lost by fermentation.

Milk Powder.—Moisture is estimated by drying 1 to 2 grammes in a basin to constant weight, and the ash is estimated in the same portion.

The estimation of fat should be made by the Gottlieb method, 0.6 to 0.7 gramme being weighed out and water sufficient to make up to 5.15 grammes; after

the addition of the alcohol, the solution may be warmed if necessary to effect complete solution, and cooled before the ether is added.

For the estimation of nitrogen by Kjeldahl's method about 1 gramme should be taken.

For other estimations 10 grammes may be dissolved in water, and made up to 100 c.c. after heating and cooling.

CHAPTER IV

THE APPLICATION OF ANALYSIS TO THE SOLUTION OF PROBLEMS

The Detection of Adulteration.—The principal forms of adulteration of milk are the addition of water and the removal of cream.

The detection of water is based on the reasoning that while the water natural to milk contains solids not fat, added water is free from these. The amount of solids not fat is nearly though not quite constant, and rarely falls below 8.5 per cent. or rises much above 9.2 per cent.; numerous cases, however, are on record of solids not fat below 8.5.

The removal of cream is detected by a deficiency in the fat; this varies much more than the solids not fat, but comparatively rarely falls below 3.0 per cent.

The probability of samples falling below 8.5 per cent. of solids not fat, and 3.0 per cent. of fat is indicated by the following Table, which gives the number of samples per 100,000 which may be expected at each percentage named; it is assumed that each sample represents a churn of milk, *i.e.* that the milk is the mixed product of several cows.

TABLE VI

Percentage of Solids not Fat	Number of Samples	Percentage of Fat	Number of Samples
8.4 to 8.5	1892	2.9 to 3.0	370
8.3 to 8.4	242	2.8 to 2.9	209
8.2 to 8.3	27	2.7 to 2.8	87
8.1 to 8.2	22	2.6 to 2.7	37
8.0 to 8.1	8	2.5 to 2.6	16
Below 8.0	2	Below 2.5	13

By Clause 4 of the Sale of Food and Drugs Act, 1899, the President of the Board of Agriculture is empowered to lay down limits below which a presumption is raised that milk is not genuine, and he has fixed 8.5 per cent. of solids not fat and 3.0 per cent. of fat. The effect of this is that the onus of proving that milk taken under the Sale of Food and Drugs Acts falling below these limits is genuine lies on the vendor, and for most practical purposes milk below these limits is taken as adulterated.

It is generally, though not invariably, found that, in milk falling below 8.5 per cent. of solids not fat, the deficiency lies chiefly on the milk sugar, and that the proteins and ash are normal; a percentage of total nitrogen above 0.5 and a percentage of ash above 0.7 in a milk below 8.5 per cent. of solids not fat will afford strong evidence that the milk is genuine, while figures for total nitrogen and ash low proportionately to the solids not fat will strengthen the conclusion that the milk is watered.

There appears to be no chemical means of distinguishing between fat naturally low and fat lowered by the abstraction of cream; the most numerous instances of fat below 3.0 per cent. naturally occurring have been found in April, May, June, and July, and they are especially rare in October, November, and December.

The percentage of added water may be calculated by the formula:

$$\text{Added water} = \frac{8.5 - S}{8.5} \times 100. \quad (S = \text{solids not fat.})$$

A formula which gives a nearer approach to the probable amount is:

$$\text{Added water} = \frac{36 - (G + F)}{36} \times 100.$$

(G = degrees of gravity. F = fat.)

The percentage of added water may be calculated from the aldehyde figure (A) by the formula :

$$\text{Added water} = \frac{20 - A}{20} \times 100.$$

The amount of cream abstracted may be calculated by the formula :

$$\text{Cream abstracted} = \frac{3 - F}{3} \times 100. \quad (F = \text{fat.})$$

This gives the minimum percentage of cream abstracted, and the more probable amount is obtained by substituting the average percentage for the month as given in chap. i. (p. 3) for 3 in both places.

Detection of Preservatives. *Boric Acid.*—To a little milk add a few drops of phenolphthalein, and caustic soda solution drop by drop till a faint pink colour is produced ; place some of the milk in two test-tubes, dilute one with an equal volume of water, and the other with a *neutral* 50 per cent. solution of glycerine ; in the absence of boric acid the two tubes will have almost the same colour, in its presence the glycerine tube will be the lighter, and usually white.

As an alternative method the milk or its ash may be made distinctly, but not strongly, acid with hydrochloric acid and a piece of turmeric paper dipped into the solution ; on drying, the paper turns pink in the presence of boric acid, and is turned a greenish black by alkalies.

Estimation of Boric Acid.—To 10 c.c. of milk add at least 5 c.c. of phenolphthalein solution ; raise to the boil, and neutralise with $\frac{N}{10}$ alkali while still boiling ; the pink colour produced when neutral is faint, though quite distinct. Add 8 to 10 c.c. of glycerine, and run in the alkali till a pink colour appears, boiling at this stage being unnecessary ; the quantity of $\frac{N}{10}$ alkali used, corrected for the acidity of the glycerine, multiplied

by 0.062 gives the percentage of boric acid (calculated as H_3BO_3).

Formaldehyde.—Dilute a little milk with an equal bulk of water in a test-tube ; pour carefully down the side of the tube a little 90 per cent. commercial sulphuric acid ; a bluish colour is developed at the junction of the acid and milk in the presence of formaldehyde. This blue colour may also be observed during the estimation of fat by the Gerber process (p. 17).

Salicylic and Benzoic Acids.—These acids give a violet colour with ferric chloride, and are best tested for by extracting the filtrate produced by treating milk with acid mercuric nitrate (p. 31) with ether, shaking the ether with a little water to which a drop of phenolphthalein solution has been added, and dropping in dilute caustic soda solution, till the water after shaking just turns pink ; after discharging the pink colour with very dilute acid and adding a little ferric chloride solution, the presence of salicylic acid is shown by a violet colour and of benzoic acid by a buff precipitate.

Hydrogen Peroxide.—Mix the sample with a little fresh milk, and add a small amount of para-phenylene-diamine or ortol ; a blue or red colour will be developed if hydrogen peroxide be present.

Reaction of Milk with Hydrogen Peroxide.—Fresh milk when treated with a little para-phenylene-diamine or ortol (a photographic developer) and a drop of hydrogen peroxide gives a deep blue (with the diamine) or a brick-red (with ortol) coloration within a few seconds. Milk heated above 80° C. remains white.

The Cause of Poor Milk.—The detection of added water and of a deficiency of cream would be an obvious explanation of the cause of milk being poor. If it is normal in composition, but very white, and the fat separated in the Gerber process is nearly free from colour, this would show that the poverty of the milk had been fallaciously inferred from its lack of colour ; if this is not the case, the test given above

should be applied, and the soluble albumin estimated (p. 35); a deficiency of albumin below 0.35 per cent., or the non-production of colour with para-phenylene-diamine or ortol will show that the milk has been heated, and as the cream rises very slowly on heated milk, the milk has been called poor because cream is not apparent in a short time. Occasionally a sample called poor turns out to contain a high percentage of fat; this would show that the milk has been standing long enough for the cream to separate and it is then divided into a rich and a poor portion.

A not unusual practice is for the servants in a household to pour off a portion of milk from a can which has stood some time for their own consumption, and remove the cream, and to send the rest of the milk, thus impoverished, for the consumption of the other members of the household.

The Cause of Sweet Milk.—Milk is sometimes alleged to be sweet. Cane sugar may be detected by adding to 10 c.c. of milk 0.1 gramme of resorcinol and 1 c.c. of strong hydrochloric acid; on standing in boiling water for five minutes a red colour is produced if cane sugar be present; it may be estimated by Harrison's method (p. 47). If all the figures for solids not fat, ash, sugar, and proteins are equally high, the milk has simply been concentrated, usually by boiling; the solids not fat have been found as high as 17.5 per cent. in a case of this kind.

The Cause of High Colour.—If the colour is yellow and the fat separated by the Gerber process is very much darker than usual, and the cream separating is much yellower than the skim-milk, the high colour is natural.

If this is not the case artificial colours should be tested for; annatto is detected by making the milk alkaline with sodium bicarbonate, immersing a strip of filter-paper in it, and allowing to stand till next day; in the presence of annatto the strip is stained brownish. Coal-tar dyes of the azo group give a pink colour when

a mineral acid is added to milk, and this is usually seen in the Gerber test. Other artificial colours are practically never used.

A pink colour is generally due to blood; to detect

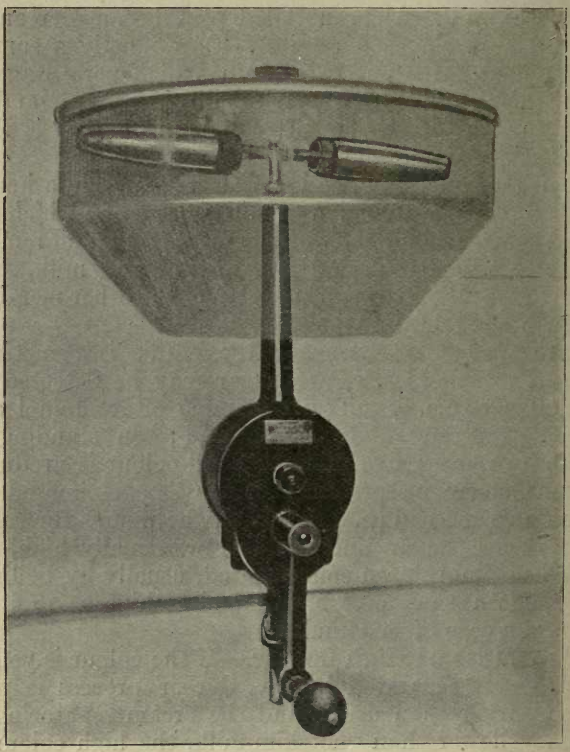


Fig. 28.—High-speed Centrifuge.

this the milk is warmed to 50° C., and separated in a high-speed centrifuge (Fig. 28); a bright red deposit at the bottom of the tube may be taken as blood; the microscopic appearance of the corpuscles is given in Fig. 29.

The Cause of Sour Milk.—Practically the only cause of milk turning sour is the formation of lactic acid by the action of micro-organisms. Milk curdles on boiling when the acidity reaches about 33°, and spontaneously in the cold when the acidity is about 80°; milk curdled by boiling generally contains somewhat hard lumps of curd, and the acidity of the whey may

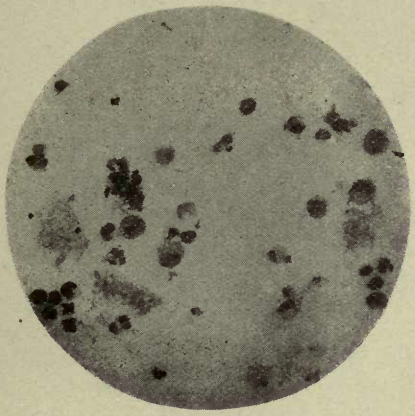


FIG. 29.—Blood in Milk

be 25° and upwards; at lower temperatures the curd is softer, and if the acidity is appreciably below 80°, this indicates that the milk has been kept warm, but not warm enough to inhibit microbial action.

Freshness of Milk.—The freshness of milk may be determined by an acidity estimation; if the acidity is more than 2° higher than the aldehyde figure, it may be assumed that a development of acidity due to the action of micro-organisms has taken place.

Many micro-organisms secrete a reductase, which decolourises methylene blue; make a solution by diluting 1 c.c. of saturated alcoholic solution of methylene blue to 100 c.c. with distilled water, add 1 c.c. of this to 25 c.c. of milk contained in a stoppered tube of little

more than 25 c.c. capacity, and keep for half an hour at a temperature of 37° C. (blood heat); if the colour disappears, the milk is not fresh.

Milk which is not fresh gives a high catalase figure,

Fig. 30.—Using Microscope.

but this is not conclusive evidence, as high catalase figures are obtained with fresh milk from cows with diseased udders.

Custards made with eggs, especially if much sugar is added, inevitably curdle if heated to too high a temperature; the liquids from such samples are usually high

in total solids (about 16 per cent.), have a low acidity, and are nearly clear, characteristics which permit of the cause of curdling being established.

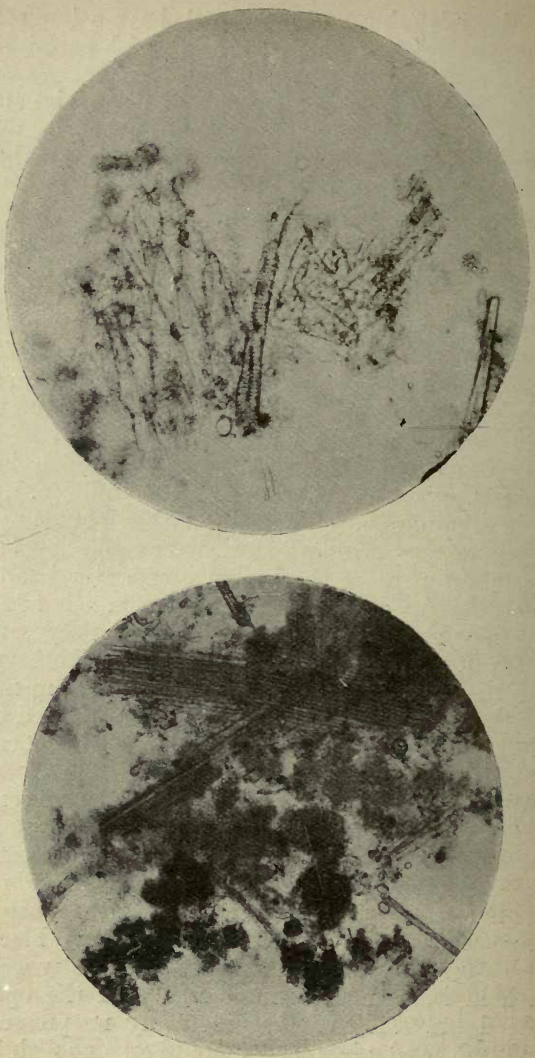
Occasionally milk is alleged to be sour because it turns blue litmus red; all milk does this, as well as turning red litmus blue, owing to the amphoteric reaction of the phosphates and citrates of the milk.

The three popular superstitions that milk can be kept warm, *i.e.* a little above blood heat for several hours without change, that custards can be heated to any temperature, and that blue litmus can be used as a test for sour milk, though all fallacious, are responsible for many allegations of sour milk. The peptonisation of milk with powders which are insufficiently alkaline or which have been kept too long may cause it to curdle, and to be thought to be sour.

The Cause of Unusual Taste.—Milk on boiling acquires a taste, and the tests for heated milk (p. 54) and soluble albumin (p. 35) will show this. Mixture with dirty water may give an evil taste to milk, and usually the establishment of the presence of water is all that can be done to explain this; if an alkali is added to milk, the taste is soapy and the smell fishy, and an increase in ash and its strongly marked alkalinity will detect the cause. If the taste is due to a fermentation other than the normal lactic one, or to the food of the cattle, it is usually difficult to detect the cause by chemical analysis.

An unpleasant taste may be due to the presence of urine, accidentally or wilfully added.

Detection of Urine.—Half fill a small test-tube ($2 \times \frac{1}{4}$ is large enough) with sodium hypobromite solution (*see* Appendix); carefully fill the tube with milk so that the two liquids do not mix. Place the thumb over the tube, and invert once or twice, and then hold it, with the thumb still over the opening, upside down. Milk causes practically no pressure on the thumb, and gives not more than one-fifth of its volume of gas; if urine has been added, much pressure



FIGS. 31, 32.—Vegetable Matter in Milk.

is developed, and the liquid squirts out ; milk containing 1 per cent. of urine yields about two-fifths the volume of gas, while 5 per cent. causes an evolution of gas equal in volume to the milk taken. Lobeck's catalase tubes (p. 39) may be used.

The Cause of Dirty Milk.—Dirty milk almost invariably deposits a sediment on standing or centrifuging ; the milk is carefully decanted, the sediment washed with water, and allowed to settle again and examined under the microscope (Fig. 30). Sharply defined vegetable cells (Fig. 31), indicate that the finer particles of the food given to the cattle probably at milking time have fallen in ; less well defined vegetable cells (Fig. 32), stained yellowish are probably derived from fæcal matter ; small hairs and various fibres (cotton, wool, &c.) show the presence of household dust ; transparent irregular particles which do not polarise are quartz, and are due to road dust ; this latter also gives a strong reaction for iron on treating the sediment with hydrochloric acid, diluting, and testing with potassium ferrocyanide which gives a blue colour with iron salts.

Control of Milk Prescriptions.—To control the preparation of milk mixtures made up for infant feeding from prescriptions, it is necessary to estimate rapidly fat, proteins, milk sugar, and added sugar. The problem is often simplified by all the materials used being available for analysis, and by determining fat (by the Gerber method), specific gravity, and aldehyde figure, and calculating the solids not fat, in the samples and in the milk from which the samples were made, the four determinations required can be obtained with sufficient accuracy for control purposes.

By estimating the ratio of solids not fat to aldehyde figure in the milk used, and multiplying the aldehyde figure of the mixture by this ratio, the amount of solids not fat derived from the milk is obtained, and the difference between the actual amount and this gives the added sugar. With an unknown milk mixture the ratio 0.45 may be assumed. The method does not,

of course, distinguish between added cane sugar or added milk sugar, but when it is known what the added substance is, an estimation of sufficient accuracy is obtained.

Detection of Adulteration of Cream.—As there is no standard for cream, it may contain any percentage of fat, and still be cream; there is a practical standard of "thickness" which the purchaser mentally estimates, and judges the value of the cream thereby. Artificial thickening is sometimes resorted to, and gelatinised starch, gelatine, "viscogen" (a solution of lime in cane-sugar syrup), condensed milk or milk powder, and collagens are added.

Starch is detected by the blue colour produced on adding a solution of iodine in potassium iodide.

Gelatine is found, if present, by diluting the cream with water and adding a little acid mercuric nitrate solution (*see* Appendix); the filtrate, if gelatine is present, is usually turbid, and gives a precipitate on the addition of a saturated solution of picric acid.

Viscogen raises the percentage of lime in the ash; the lime on an average amounts to 22 per cent. of the ash, and its ratio to phosphoric acid (CaO to P_2O_5) is 1 : 1.3. Viscogen raises not only the percentage in the ash, but also the ratio to the solids not fat. A small cane-sugar reaction may be obtained, and the percentage of sugar polarised as milk sugar will exceed 52.5 per cent. of the solids not fat.

Condensed milk or *milk powder* may be detected by the solids not fat being found in much greater proportion than that given as corresponding to the fat found in Table V; the percentages of ash, milk sugar, and proteins, and the aldehyde figure will bear the same proportion to the solids not fat as found in milk. Clotted or Devonshire cream, however, is concentrated during its preparation, but its physical appearance differs from that of raw cream, and it does not give the reactions with hydrogen peroxide given on p. 54.

Collagens are difficult of detection; they raise the

percentage of solids not fat, and give the same reaction as cane sugar with resorcinol (p. 55), but by Harrison's method no cane sugar is shown.

Preservatives are detected in the same way as for milk. For the estimation of boric acid, the cream should be diluted with an equal weight of water.

Adulteration of Skim-milk.—The President of the Board of Agriculture has fixed 9.0 per cent. as the limit for total solids in skim-milk. Percentages below this are presumed to be caused by the addition of water.

Rennet is sometimes added to skim-milk, and even to whole milk, usually with the idea of causing curdling when the milk is warmed. Its presence may be inferred if the milk curdles on warming to 40° C., and the acidity is less than 25°; the whey on neutralising to an acidity of 12° will cause fresh milk to curdle at 40°, and the amount of lime in the whey does not exceed 0.06 per cent.

Detection of Foreign Fats in Milk and Cream.—By means of an emulsifying apparatus, foreign fats (margarine fat, cocoa-nut oil) are mixed with separate milk and the product sold as milk or cream. The casein should be precipitated from a considerable amount of milk (p. 35), dried, extracted with ether, and the fat examined as butter fat (pp. 67 *et seq.*).

CHAPTER V

THE ANALYSIS OF BUTTER

Estimation of Water.—Weigh a small round basin (Fig. 33) about 3 in. in diameter, containing a small rod; place 5 to 10 grammes of butter therein, and weigh again; heat the basin over a very small flame, or on a sand-bath, and stir *constantly* till frothing has ceased, cool, and weigh again. The loss of weight indicates water, and this multiplied by 100 and divided by the weight of butter taken gives the percentage. The flame should be of such a size that the butter takes at least a minute to become melted.

As an alternative method, about $2\frac{1}{2}$ grammes of butter may be weighed in a flat-bottomed basin, just melted in the water-oven, and $1\frac{1}{2}$ c.c. strong alcohol mixed with the melted fat; the basin is placed in the water-oven for two hours, cooled, and weighed. The loss of weight gives the water.

Estimation of Curd and Salt.—Wash the fat from the basin after driving off the water by nearly filling it with ether or amyl alcohol, and carefully decanting the liquid after the solid portion has settled, and repeating this four times; if amyl alcohol is used, it should be hot; the residue is dried in the water-oven for two hours and weighed after cooling. This represents curd and salt (if present).

Extract the salt from the curd with hot water, and filter the solution; wash the residue and the filter, and cool the filtrate; add a few drops of potassium chromate solution, and titrate with $\frac{N}{10}$ silver nitrate solution till a faint red colour just appears; each cubic centimetre used is equal to 0.00585 gramme salt.

Estimation of Casein.—Extract another portion of curd with dilute ammonia till no lumps are left; filter and wash the residue; add dilute acetic acid till a white precipitate falls, and collect this in a weighed Gooch crucible

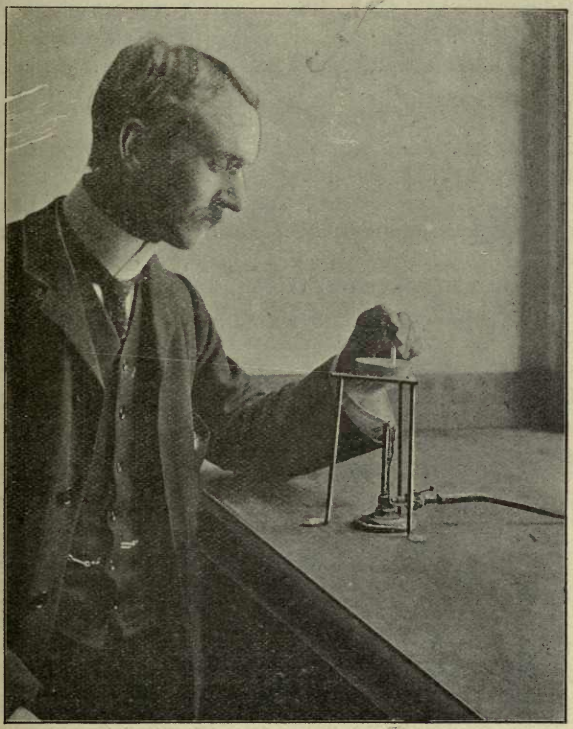


FIG. 33.—Water Estimation.

or on tared filter-papers, as on p. 35. Extraction and ignition may be omitted. The precipitate is casein.

Tests for Preservatives.—Boric acid may be detected by melting the butter at a low temperature, and testing the aqueous portion with turmeric paper as directed on p. 53.

If found, the estimation is carried out by weighing 25 grammes of butter, adding 25 c.c. of water, melting the butter at a low temperature, and stirring well; the aqueous portion is allowed to settle, and 20 c.c. are withdrawn, and placed in a small beaker and boiled; at least 10 c.c. of phenolphthalein solution (*see* Appendix) is added, and the solution titrated while still boiling with $\frac{N}{2}$ caustic soda solution (*see* Appendix) till a faint pink colour occurs; the reading of the burette is noted, 12 c.c. glycerine are added, and the solution titrated again (boiling is not necessary) till a pink colour appears; the difference between the reading of the burette and the first reading, corrected for the acidity of the glycerine, multiplied by 0.031, will give the weight of boric acid (as H_3BO_3), and this multiplied by $5 + 0.05$ W (W = percentage of water) will give the percentage of boric acid. Where the percentage of water is not estimated the figure 5.65 may be used for $5 + 0.05$ W, and as an approximate figure the number of cubic centimetres of $\frac{N}{2}$ soda solution used between the first and second titrations may be multiplied by 0.17.

Sulphites are detected by the smell of sulphurous acid given off on acidifying the aqueous portion; formaldehyde will give its characteristic reaction (p. 54) if a little of the aqueous portion is added to milk; fluorides are detected by adding a solution of calcium chloride to the aqueous portion, filtering, igniting the precipitate, taking up with dilute acetic acid, and treating the insoluble portion with strong sulphuric acid in a platinum dish over which is inverted a glass plate coated with beeswax through which one or two lines are scratched; on warming the sulphuric acid, hydrofluoric acid is given off in the presence of fluorides, and this etches the portions of the glass exposed by scratching through the beeswax, and the marks are visible on melting and wiping off the beeswax.

The Examination of the Fat.—Butter fat is of peculiar composition, consisting of complex glycerides containing lower fatty acids, chiefly caproic and butyric acids; these are characterised by being soluble in water, and volatile with steam, while the fatty acids of almost all other fats are insoluble and non-volatile; furthermore the presence of the lower fatty acids in the glycerides causes them to have a softer consistency than if only the insoluble acids were present, and a comparatively small amount of the acids of the oleic and more unsaturated series is present. To obtain a fat of the consistency of butter without the lower fatty acids, a larger amount of acids of the oleic, &c., series must be present. The presence of the lower fatty acids gives a high specific gravity to the glycerides, and causes them to crystallise badly.

In addition to these facts on which the broad principles of butter analysis are based, certain vegetable oils, especially sesamé oil, give characteristic reactions, and it has been recommended that by international agreement all margarine shall legally be made to contain 10 per cent. of sesamé oil.

The addition of margarine to butter may be detected by—

(*a*) A lowered proportion of volatile acids;
(*b*) A lowered proportion of soluble and increased proportion of insoluble acids;
(*c*) An increased mean molecular weight;
(*d*) A decreased density;
(*e*) A more marked crystallisation;

properties all chiefly depending on the lowering of the amount of caproic and butyric acids in the glycerides, and—

(*f*) An increased iodine absorption;
(*g*) An increased refractive index;

properties chiefly depending on the increase of the unsaturated acids in the glycerides; to those may be added—

(*h*) A turbidity of the fat on melting at a low

temperature, a property which depends on the fact that mixing margarine with butter often causes overworking, which gives rise to turbidity.

Owing to the natural variations of the composition of butter fat, and to a less degree of the composition of adulterants, the detection of small quantities of margarine is difficult, and unless sesamé oil can be found, impossible in minimal amounts ; as only three principles underlie all the methods, one of which has little value, a multiplication of tests does not greatly assist.

Preparation of the Fat for Analysis.—Place 20 to 50 grammes of butter in a small beaker, and put this in the water oven till melted ; observe the fatty layer, whether clear or turbid ; pour as much as possible of the fat into a dry filter, taking care that none of the aqueous portion accompanies it, and filter in the water oven, collecting the clear fat in a small beaker.

Estimation of the Volatile Fatty Acids. *Reichert-Wollny Process, Polenske's Modification.*—Place a flask of 300 c.c. capacity on one pan of a balance and tare it ; add 4.5 grammes to the weights, and run in the melted fat till the flask is weighed down, and place a further 0.5 gramme weight on the other pan ; continue the addition of the fat cautiously, till the weight is exact, if necessary removing a surplus with a small pipette. It is not necessary to wait for the fat in the flask to cool before making the final adjustment, as the error involved in weighing warm fat is within the limits of error of the final titration, nor is it necessary to weigh more accurately than to 0.005 gramme. Weigh in 20 grammes of glycerine.

Add 2 c.c. of a 50 per cent. solution of caustic soda (*see* Appendix), preferably from a special measuring apparatus (Fig. 34) ; heat the mixture over a naked flame till the turbid liquid suddenly becomes clear ; allow it to cool a little and add 100 c.c. of hot water which has been previously boiled for at least fifteen minutes, and when all the resulting soap is dissolved 0.1 gramme of powdered pumice which has been sifted

through muslin and ignited, and 40 c.c. of sulphuric acid solution (*see* Appendix); attach the flask by means of a cork to a bulb tube attached to a condenser, the dimensions of which are given in the sketch

Fig. 34.—Caustic Soda Apparatus.

(Fig. 35). Support the flask on a piece of asbestos card in which is cut a hole 5 c.m. in diameter and heat with a very small flame, till the fatty acids float in a clear layer on the surface of the liquid; then turn up the flame to such a height that 110 c.c. distil in from eighteen to twenty-two minutes. Collect 110 c.c., turn out the

flame, and replace the flask by a 25 c.c. cylinder; cool the flask for ten minutes in water at 10° C., mix the distillate, and filter through a dry filter; use the first few c.c. to wash out a 100 c.c. flask, and collect exactly 100 c.c. of the filtrate, and transfer this to a beaker, add a little phenolphthalein and titrate with $\frac{N}{10}$ baryta, strontia, or soda solution till a pink colour appears; pour this back into the flask, and again into the beaker, and if the colour is discharged continue the titration. From the number of cubic centimetres used, subtract the figure obtained in a blank experiment, and multiply the result by 1.1 to obtain the Reichert-Wollny figure.

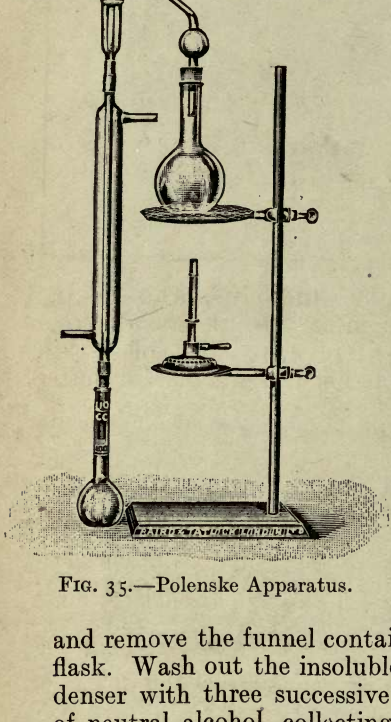


FIG. 35.—Polenske Apparatus.

Wash out the condenser with two successive quantities of 9 c.c. each of cold water, collecting this in the cylinder and using it to wash out the flask, and pouring it through the filter; reject this filtrate, and remove the funnel containing the filtrate to a clean flask. Wash out the insoluble fatty acids from the condenser with three successive quantities of 10 c.c. each of neutral alcohol, collecting each in the cylinder, and pouring it through the filter; titrate the combined filtrates with $\frac{N}{10}$ alkali after adding a little phenolphthalein; the number of c.c. of $\frac{N}{10}$ alkali used gives the Polenske figure.

The Polenske figure varies with the Reichert-Wollny figure, and the following Table shows the relation :

TABLE VII

Reichert-Wollny Figure	Polenske Figure	
	Mean	Maximum
32	3.2	3.7
31	3.0	3.5
30	2.8	3.3
29	2.7	3.2
28	2.6	3.1
27	2.4	2.9
26	2.3	2.8
25	2.1	2.6
24	2.0	2.5
23	1.9	2.4
22	1.8	2.3
21	1.7	2.2

Estimation of Soluble and Insoluble Fatty Acids and Mean Molecular Weight. — Weigh a glass flask, which has been previously well boiled with caustic alkali solution ; add about 4 grammes of butter fat, and weigh again. Run in 50 c.c. $\frac{N}{2}$ alcoholic soda solution (*see* Appendix), attach the flask to an upright condenser, and boil for a quarter of an hour ; add a few drops of phenolphthalein solution and titrate with $\frac{N}{2}$ hydrochloric acid till the pink colour is discharged. As the alcoholic soda solution alters in strength it must be checked against the $\frac{N}{2}$ hydrochloric acid, and the value in terms of the acid solution obtained of 50 c.c. of alcoholic soda ; from this subtract the volume of $\frac{N}{2}$ hydrochloric acid used in titrating after saponification, and multiply by 2.805 and divide by the weight of butter taken ; the figure obtained will be the percentage of potash required for saponification.

Wash out the alcoholic solution into a large basin, and evaporate the alcohol on the water-bath; add enough hot water to make the bulk up to 150 to 200 c.c. and then add sufficient $\frac{N}{2}$ hydrochloric acid solution to make with the volume already used in the titration 1 c.c. more than the quantity equal to the 50 c.c. alcoholic soda added; heat on the water-bath till the insoluble fatty acids float on the surface in a clear layer, and filter through a wet filter; wash out the basin with hot water till the fatty acids are transferred to the filter, and wash them well on the filter, stirring them up with the jet of water; at least a litre of water is required for washing. With a good filter the fatty acids do not run through; when washed, allow all the water to run out, and wash the filter with hot alcohol, collecting the filtrate in a weighed flask, till all the fatty acids are removed; evaporate the alcohol, and dry in the water-oven till the weight is constant; this will give the insoluble fatty acids.

To estimate the soluble fatty acids, add a little phenolphthalein solution to the filtrate from the insoluble fatty acids, and titrate with alcoholic alkali till a pink colour appears; from the volume used calculate the equivalent of $\frac{N}{2}$ acid, from this subtract the 1 c.c. added in excess of the soda added, multiply by 4.4, and divide by the weight of butter taken; this will give the soluble fatty acids in terms of butyric acid.

Avé-Lallemant Method.—Four grammes of fat are saponified and neutralised as directed above, and the alcohol evaporated; 20 c.c. of water are added, and the saponified fat evaporated to dryness. About 350 c.c. of hot water are added, and the soap solution transferred to a 500 c.c. flask, which is placed on a water-bath. 100 c.c. $\frac{N}{5}$ barium chloride solution are added with constant shaking, and the flask is left for fifteen minutes on the water-bath; it is then cooled,

and the solution made up to 500 c.c. and filtered; 200 c.c. of the filtrate is raised nearly to the boiling-point, and dilute sulphuric acid added till no further precipitate is obtained, the precipitate collected on a filter, washed with water, alcohol, and ether, ignited and weighed; the difference between the weight and that of the barium sulphate obtained from 40 c.c. of the barium chloride solution, multiplied by 1643.5 and divided by the weight of fat taken, gives the insoluble baryta value (*b*). The saponification value is calculated in terms of barium oxide by multiplying the percentage of potash required for saponification (*see above*) by 13.68 (*a*), and the difference between *a* and *b* will give the soluble baryta value (*c*).

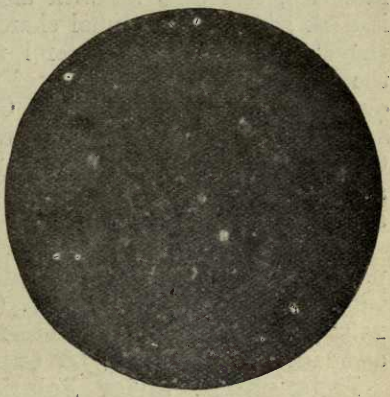


FIG. 36.—Butter.

In genuine butters the value of $b - (200 + c)$ is always negative, and varies from -0.7 to -23.8; cocoa-nut oil, margarine, and other fats give positive values.

The soluble baryta value of butter usually lies between 50 and 65, but may be occasionally higher; cocoa-nut oil gives nearly the same figure, but the soluble baryta value for other fats does not exceed 10.

Phytosteryl Acetate Method.—Fifty grammes of the clear melted fat are shaken with 75 c.c. of warm 95 per cent. alcohol; the alcohol is cooled and decanted, and a further quantity of 75 c.c. of alcohol is added, and the process repeated.

The fat is saponified with 1 c.c. of 50 per cent. caustic soda solution, and the bulk of the alcohol evaporated in a basin; 2 grammes of sodium bicarbonate and

2 or 3 grammes of kieselguhr are added, and the mass evaporated to dryness, placed in an extraction thimble, and extracted with petroleum ether in a Soxhlet. Evaporate the solution, add 5 c.c. of $\frac{N}{2}$ alcoholic soda solution, and evaporate to dryness, adding a little sodium bicarbonate before the solvent has completely evaporated. Extract with petroleum ether, and evaporate this, and take up with alcohol; if the solution is dark add a little animal charcoal and filter. Allow the cholesterol or phytosterol to crystallise, and to the crystals add 2 or 3 c.c. of acetic anhydride, and evaporate on the water-bath. Crystallise the acetates several times from alcohol and take the melting-point. Cholesteryl acetate melts at 113.2°–114.6° (corr.) and a higher melting-point shows presence of phytosteryl acetate.

Estimation of Density.—Follow the directions on p. 10, with the following modifications: weigh the tube full of water at 37.8° C. (100 F.) instead of at 15.5° C.; dry the tube before filling it with the fat, and take the density at 37.8° C. instead of at 15.5° C.

Examination under Polarised Light.—Place a small portion of the butter (not butter fat) on a microscope slide, and press down a cover-glass thereon; examine with a microscope furnished with a polariscope, using a 1 in. or $\frac{1}{2}$ in. power, focus with the Nicols parallel, and then cross them, shielding the slide from light except that which has passed through the polariser. Genuine butter appears nearly uniformly dark, while crystalline fats show a more or less well-marked lighting in portions of the field.

Old butters, especially those which have been submitted to vibrations, and butters prepared by processes in which the cream is churned soon after heating and cooling may show (Fig. 36) a somewhat crystalline appearance, but generally this is due to margarine (Fig. 37); this test though very rapid may not be reliable.

Estimation of Iodine Absorption.—Weigh about 0.4 to 0.5 gramme of fat in a stoppered bottle, dissolve in 10 c.c. of chloroform, and add 20 c.c. of Wijs' iodine solution (*see* Appendix). At the same time mix 10 c.c. of chloroform with 20 c.c. of iodine solution, and place both bottles in a dark place for half an hour. Add 15 c.c. of a 10 per cent. solution of potassium iodide solution to each, and about 200 c.c. of water, and titrate with $\frac{N}{10}$ sodium thiosulphate solution (*see* Appendix) till the colour on shaking is removed from both aqueous and chloroformic solutions; a little starch solution may be added when the colour is very pale, and the titration carried on till all blue has disappeared, but its use is not absolutely necessary.

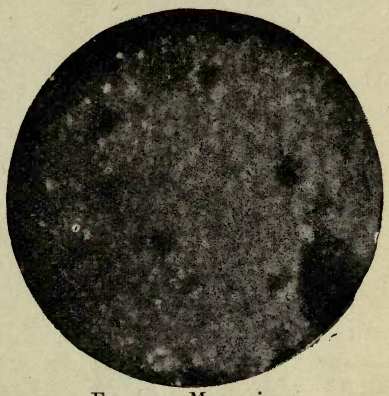


FIG. 37.—Margarine

The number of c.c. of thiosulphate multiplied by the value of 1 c.c. in terms of iodine used in the blank experiment gives the total amount of iodine added to the fat; the number of c.c. of thiosulphate similarly multiplied used in the actual experiment gives the weight of iodine not absorbed by the fat, and the difference between these two gives the quantity absorbed, and this multiplied by 100 and divided by the weight of fat taken is the iodine absorption.

Determination of Refractive Index.—The Zeiss butyro-refractometer is employed for the determination of the refractive index; it consists of two water-jacketed prisms, between which the substance is placed, a mirror to reflect light through these, an eye-

piece with a scale in it by which the refractive index is read off, and a thermometer for observing the temperature. An apparatus for providing a stream of water of constant temperature can be used, or in default

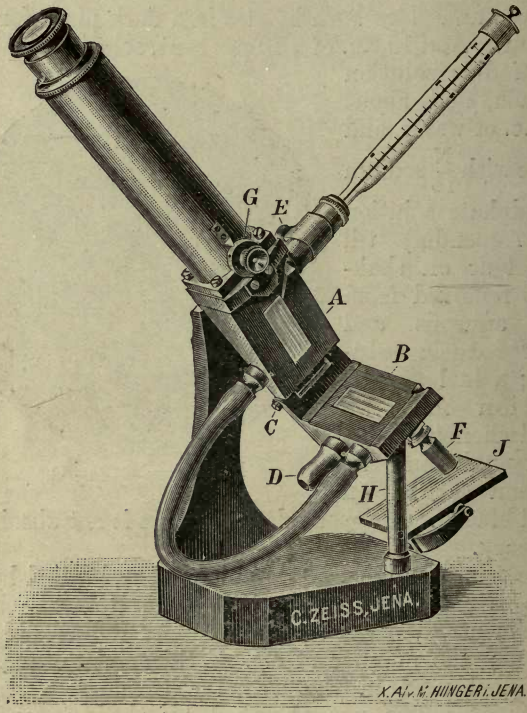


FIG. 38.—Refractometer.

of this a stream of water warmed to the required degree can be run through the jacket by india-rubber tubes from any fair-sized vessel ; though it is an advantage to use water at a constant temperature, the cooling of a large bulk of water is sufficiently slow to keep the prisms at practically a constant temperature during the reading.

To make an observation, take the refractometer (Fig. 38), out of its case, stand it up, screw it in the thermometer, and connect the india-rubber tubes carrying the water to the inlet and outlet tubes of the

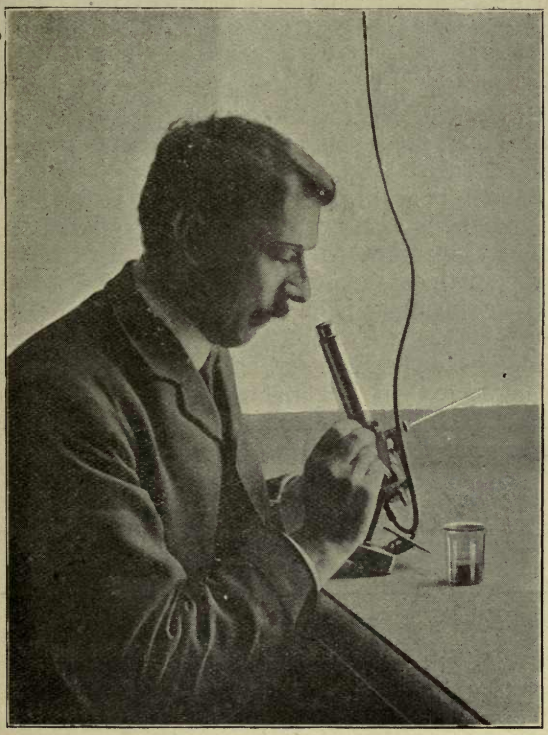


FIG. 39.—Refractometer

jacket. Turn the milled head, and open and throw back on to its support the lower prism; see that both glass surfaces are clean (a cloth dipped in alcohol, followed by a dry soft cloth is best for cleaning), and place a drop of the melted fat in the centre of the surface of the lower prism (Fig. 39), and close it.

Arrange the mirror to reflect either daylight or the light of a lamp through the prisms, and observe the point on the scale where the dark shadow comes, focusing the eye-piece if necessary; make two or three observations till the position of the shadow is constant, and read the temperature at the same time. The thermometer may be graduated either in centigrade degrees, or in the normal reading for butter fat at the temperature of the water; if of the former kind, both readings are noted, and the refractometer readings corrected to a standard temperature by multiplying the difference between the observed temperature and the standard temperature by 0.55, and adding or subtracting the result, as the temperature is higher or lower than the standard, to the refractometer reading. If the thermometer is of the latter type, the observed reading of the refractometer is subtracted from the reading of the thermometer, and the result is expressed in degrees less than the standard.

Table VIII gives the standard readings for each five degrees centigrade.

TABLE VIII

Temperature	Scale Divisions
25	52.5
30	49.8
35	47.0
40	44.2
45	41.5

The average figure for butter is 46° at 35° C. or a little below, and margarine gives about 54°. Genuine butter sometimes gives a reading higher than 47°, and the limits found are 43.5° to 49°.

A standard solution (*normal flüssigkeit*) is supplied with the instrument, and the scale should be adjusted from time to time with this; a point is marked on the

scale where the standard solution should read, and a key is provided for adjusting the scale to this.

Detection of Sesamé Oil.—Add to 10 c.c. of the melted butter 0.1 c.c. of a 2 per cent. alcoholic solution of furfural, add 1 c.c. of strong hydrochloric acid, shake well and add 10 c.c. of chloroform. A crimson coloration of the aqueous layer indicates sesamé oil. Sometimes coal-tar colours are added to the butter which give a red colour with hydrochloric acid alone; make sure of the absence of these by testing a little of the butter with hydrochloric acid. If these are present use the fatty acids (p. 72) for the test.

If furfural is not available dissolve 0.1 gramme of sugar in 5 c.c. of hydrochloric acid, and shake with 10 c.c. of melted fat. This will also give a crimson colour with sesamé oil.

Detection of Cocoa-nut Oil. *Hinks' Method.*—Dissolve 5 c.c. of fat in 10 c.c. of ether, in a corked test-tube, and pack this in ice; after thirty minutes pour the whole mass on a filter, and evaporate the filtrate; take up the fat with 3 to 4 c.c. of 96 to 97 per cent. alcohol, boil when the fat should all dissolve, and cool to 5° C. for fifteen minutes, and filter this; cool the filtrate to 0° C. for two to three hours. Place a little of the cooled solution on a well-cooled slide, and examine rapidly with a $\frac{1}{2}$ or $\frac{2}{3}$ in. objective. Butter crystallises in round globular masses, while cocoa-nut oil gives characteristic feathery crystals, easily recognised after a little practice; other fats such as lard give somewhat similar crystals.

The Application of Analysis to the Solution of Problems. *The Detection of Adulteration.*—The President of the Board of Agriculture, under his powers authorised by Clause 4 of the Sale of Food and Drugs Act, 1899, has laid down the limit of 16 per cent. of water in butter, and any quantity above this amount is presumed to have been added. Occasionally water has been worked into butter to add to its weight, but a more frequent occurrence of excess of water occurs in

milk-blended butter; in the latter the "curd" will be in the proportion of 1 part for each 10 parts of water, in the former much less.

A washed butter may be distinguished from an unwashed one by containing less than 1 part of curd to 10 parts of water, usually only about 0.5 part; unwashed butter contains about 1 part; a larger percentage of curd indicates that the butter was churned from very sour cream, and will not keep well, or that it has been adulterated with casein. A higher percentage of casein than 0.5 indicates the presence of added casein; this form of adulteration is not uncommon. Cane sugar or honey is occasionally added; the test given on p. 54 applied to the aqueous portion obtained on melting out will detect this. The percentage of salt should not exceed 3 in a mild salt butter, but may go up to 5 per cent. or more in other samples. Irish pickled butters are high in water and in salt.

The Preservatives Committee of the Local Government Board recommended that no preservative except boric acid be allowed in butter, and that only to the extent of 0.5 per cent.; this has not yet been legalised.

The detection of margarine is more difficult; the Butter Regulations Committee of the Board of Agriculture has recommended a limit for the Reichert-Wollny figure of 24 c.c., but their recommendation has not yet been adopted.

This figure, as well as 89 as a superior limit for insoluble fatty acids, 5 per cent. as the minimum for soluble fatty acids, 22.0 per cent. for the potash required for saponification, 0.910 for the density, 42 per cent. for the iodine absorption, and 48° as a maximum Zeiss butyro-refractometer figure, may be taken as indicating the border-line of genuine butters. Certain butters, however, especially those prepared from the milk of cows exposed to cold climates and near the end of lactation, yield figures beyond these limits. The appearance on melting, and under polarised light, may

be useful as confirmatory, and a reaction for sesamé oil will establish the presence of margarine, while vegetable oils give the phytosteryl acetate test.

The average figures for butter and margarine are :

TABLE IX

	Butter	Margarine	Cocoa-nut
Reichert-Wollny figure .	29 cc.	Practically none	7
Polenske figure . .	2.5	2.0	17
Insoluble fatty acids .	87.5 %	95.5 %	85
Soluble fatty acids . .	6.0 %	None	?
Potash required . .	22.7 %	19.5%	26
Density	0.913	0.902	?
Iodine absorption . .	37	55	9
Refractometer at 35° .	46°	54°	43°

Cocoa-nut oil has a different composition from that of margarine ; but as it lowers the Reichert-Wollny figure, while at the same time raising the Polenske figure and potash absorption, and lowering the insoluble fatty acids, iodine absorption and the refractometer figure, its detection is not difficult. Positive evidence of its presence can be obtained by Hinks' method.

Application of Analysis to Buttermaking.—The acidity of the cream should be taken to see if it is properly ripened ; an acidity of 60° to 70° is usually suitable.

The fat in the butter-milk is an important item, as this represents loss ; 0.2 per cent. shows satisfactory churning, while more than 1 per cent. even with very thick cream indicates bad working ; either the ripening has not been properly done, the churning is too rapid, or the temperatures are wrong.

The amount of butter yielded by milk may be calculated by subtracting 0.1 from the percentage of fat, multiplying by 7, and dividing by 60 ; this gives the number of pounds of butter per gallon of milk.

The following Table will give the amount of butter

that may be expected from cream of any percentage of fat :

TABLE X

Percentage of Fat in Cream	Quarts of Cream Churned									
	1	2	3	4	5	6	7	8	9	10
15	.44	.87	1.31	1.74	2.18	2.61	3.05	3.48	3.92	4.35
16	.47	.93	1.40	1.86	2.33	2.69	3.16	3.72	4.19	4.65
17	.50	.99	1.49	1.98	2.48	2.98	3.47	3.97	4.46	4.96
18	.53	1.05	1.58	2.10	2.63	3.16	3.68	4.21	4.73	5.26
19	.56	1.11	1.67	2.23	2.79	3.34	3.90	4.46	5.01	5.57
20	.59	1.17	1.76	2.35	2.94	3.52	4.11	4.70	5.28	5.87
21	.62	1.23	1.85	2.46	3.08	3.70	4.31	4.93	5.54	6.16
22	.65	1.29	1.94	2.58	3.23	3.87	4.52	5.16	5.81	6.45
23	.68	1.35	2.03	2.70	3.38	4.05	4.73	5.40	6.08	6.75
24	.70	1.41	2.11	2.82	3.52	4.22	4.93	5.63	6.34	7.04
25	.73	1.47	2.20	2.93	3.67	4.40	5.13	5.86	6.60	7.33
26	.76	1.52	2.29	3.05	3.81	4.57	5.33	6.10	6.86	7.62
27	.79	1.58	2.37	3.16	3.96	4.75	5.54	6.33	7.12	7.91
28	.82	1.64	2.46	3.28	4.10	4.91	5.73	6.55	7.37	8.19
29	.85	1.70	2.54	3.39	4.24	5.09	5.94	6.78	7.63	8.48
30	.88	1.75	2.63	3.51	4.38	5.26	6.14	7.02	7.89	8.77
31	.91	1.81	2.72	3.62	4.53	5.43	6.34	7.24	8.15	9.05
32	.93	1.87	2.80	3.74	4.67	5.60	6.54	7.47	8.41	9.34
33	.96	1.92	2.89	3.85	4.81	5.77	6.73	7.70	8.66	9.62
34	.99	1.98	2.97	3.96	4.96	5.95	6.94	7.93	8.92	9.91
35	1.02	2.04	3.06	4.08	5.10	6.11	7.13	8.15	9.17	10.19
36	1.05	2.09	3.14	4.18	5.23	6.28	7.32	8.37	9.41	10.46
37	1.07	2.15	3.22	4.30	5.37	6.44	7.52	8.59	9.67	10.74
38	1.10	2.20	3.30	4.40	5.51	6.61	7.71	8.81	9.91	11.01
39	1.13	2.26	3.39	4.52	5.65	6.77	7.90	9.03	10.16	11.29
40	1.16	2.31	3.47	4.62	5.78	6.94	8.09	9.25	10.40	11.56
41	1.18	2.37	3.55	4.73	5.92	7.10	8.28	9.46	10.65	11.83
42	1.21	2.42	3.63	4.84	6.05	7.25	8.46	9.67	10.88	12.09
43	1.24	2.47	3.71	4.94	6.18	7.42	8.65	9.89	11.12	12.36
44	1.26	2.52	3.79	5.05	6.31	7.57	8.83	10.10	11.36	12.62
45	1.29	2.58	3.87	5.16	6.45	7.73	9.02	10.31	11.60	12.89
46	1.32	2.63	3.95	5.26	6.58	7.89	9.21	10.52	11.84	13.15
47	1.34	2.68	4.02	5.36	6.70	8.04	9.38	10.72	12.06	13.40
48	1.36	2.73	4.09	5.46	6.82	8.18	9.55	10.91	12.28	13.64
49	1.39	2.77	4.16	5.55	6.94	8.32	9.71	11.10	12.48	13.87
50	1.41	2.82	4.23	5.64	7.05	8.46	9.87	11.28	12.69	14.10

CHAPTER VI

THE ANALYSIS OF CHEESE

Estimation of Water, Ash, and Salt.—Place 2 or 3 grammes of cheese cut into small pieces in a small flat-bottomed basin, and keep in the water-oven for six hours; the drying proceeds better if the basin is inclined so that the fat runs off the drying cheese; weight and return to the water-oven, and weigh again at intervals of one hour till the loss is less than one milligramme per hour; the loss may be taken as water.

Pour off as much fat as possible, and macerate the residue in hot amyl alcohol; pour off the amyl alcohol as completely as possible, and ignite the residue as in determining the ash of milk (p. 12); to determine the salt make a determination of chlorine as on p. 14; each cubic centimetre of $\frac{N}{10}$ silver nitrate is equal to 0.00585 grammes salt.

Estimation of Fat.—Weigh about 2 grammes of cheese, cut into small pieces, and transfer to a Stokes tube, add 8 c.c. of water, and heat gently till the cheese is softened and disintegrated; then add 10 c.c. of hydrochloric acid, and treat as in the Werner-Schmid method (p. 29).

Estimation of Total Nitrogen.—Weigh about 1 gramme of cheese and treat by the Kjeldahl method (p. 35).

Estimation of Products of Ripening.—Weigh 10 grammes of cheese, place in a small mortar, and add 25 c.c. of boiling water; with a pestle grind up the cheese and water, and pour off the solution through a filter, collecting the filtrate in a 250 c.c. flask; repeat the treatment with 25 c.c. of boiling water till nine

portions have been used ; cool the total filtrates, make up to 250 c.c. and mix well. Evaporate 50 c.c. in a weighed basin, and weigh the solids after drying till the loss is less than 1 milligramme per hour ; ignite, and weigh the ash ; the weight of the solids less that of the ash represents the products of ripening. The difference between 100, and the sum of the water, fat, ash, and products of ripening, may be taken as unaltered paracasein.

The products of ripening may be differentiated ; to 50 c.c. of the filtrate add 5 c.c. of copper sulphate, and treat as in the Ritthausen method for the estimation of proteins (p. 34) ; the proteins estimated in this way may be termed primary products of ripening, and the remainder secondary products.

Examination of the Fat.—Dry 25 to 50 grammes of cheese till the fat runs out ; extract with ether, and wash the ethereal solution with water in a separating funnel ; remove the ethereal layer, and drive off the ether, and dry the fat till the ether is completely removed. Examine the fat as directed for butter fat ; usually a Reichert-Wollny figure is required.

The Application of Analysis to the Solution of Problems. *Detection of Adulteration.*—Practically the only adulterations of cheese consist in the removal of fat from the milk before curdling, and the addition of foreign matter.

The removal of fat may be judged if the fat is less than 45 per cent. of the dried cheese, or if the fat is less than six times the total nitrogen, both of which standards lead to practically the same result ; a large number of cheeses are made with half-skimmed milk (*e.g.* the evening's milk is skimmed, and mixed with the fresh morning's milk) ; these fail to comply with the above standards, and should be sold as half-skim cheeses ; other cheeses are made from skim-milk alone, *e.g.* Dutch cheese (though cheeses are made in Holland with whole milk also).

Another method, particularly applicable to cream cheese, is to deduce the composition of the cream (or milk) used for the preparation of the sample, as follows :

Divide the proteins by 0.3, which will give the equivalent of solids not fat.

Divide the solids not fat thus calculated by 0.104, which yields the equivalent of water in the original cream (or milk).

The sum of the water, the solids not fat, and the fat, represents the original cream, and the fat and solids not fat are calculated as percentages. To allow for loss, add on 0.25 per cent. to the percentage of the fat thus deduced.

An example will make this point clearer. A cream cheese contains 4.1 per cent. of proteins, and 49.5 per cent. of fat.

$$\begin{aligned} 4.1 \div 0.3 &= 13.7 \text{ solids not fat.} \\ 13.7 \div 0.104 &= 132.7 \text{ water.} \\ & \quad 49.5 \text{ fat found.} \\ \text{Total} \quad .. & \quad 195.9 \end{aligned}$$

$$\text{Percentage of solids not fat} = 13.7 \times \frac{100}{195.9} = 7.0$$

$$\text{Percentage of fat} = 49.5 \times \frac{100}{195.9} = 25.3 + 0.25 = 25.5$$

The quality of the cheese is then judged by the quality of the milk or cream used for its preparation.

The detection of " margarine-cheese " is accomplished by the examination of the fat ; practically the same standards as for butter may be used ; it must be remembered, however, that during the ripening the fat is slightly attacked, and the percentage of volatile acids may be somewhat lowered by this cause.

Preservatives need not be looked for in cheese.

Application of Analysis to Cheese-making. *Estimation of the curd by Lindet's Method.*—Estimate

the fat and specific gravity as previously described; to 100 c.c. of milk add 0.01 gramme of rennet powder, and keep at 42° C. till curdled; cut up the curd and allow it to settle, and strain off the whey through muslin; cool the whey to 15.5° C. and estimate the specific gravity and the fat as before.

Add the degrees of gravity and the percentage of fat of the milk and subtract the sum of the degrees of gravity and the percentage of fat of the whey; the difference divided by 3.5 will give the percentage of dry curd available for cheese-making.

In practice the whey obtained during cheese-making may be tested instead of a separate preparation of whey being made; the sample for testing should, however, be removed as early as possible.

The difference in acidity (*see* p. 15) between the milk and the whey divided by 3.5 will also give a rough estimation of dry curd.

This will give an idea of the value of milk for cheese-making. The percentage of dry curd plus the percentage of fat less 0.25 divided by 0.055 will give the number of pounds of cheese per 100 gallons of milk.*

A fermentation test is useful; plug a number of clean test-tubes with cotton-wool, and sterilise by heating to 150° C. in an air-bath for half an hour. Place 10 c.c. of the milk to be examined in one of these, and keep it at blood-heat for eighteen hours; if the precipitated curd is distended by bubbles of gas the milk will not make good cheese.

The acidity of the milk before renneting should be estimated (p. 15), and that of the whey after the curd is cut, and the whey running from the curd at intervals. As an example of the use of the acidity test, in Cheddar cheese-making the best acidity of the milk for renneting is 22°–24°; the acidity of the whey is less than this, but constantly increases; the whey should be drawn

* The factor 0.055 will require modification according to the kind of cheese made; it applies fairly well to Cheddar and Cheshire cheese.

at about 22° acidity, and the curd vatted when the whey draining off has an acidity of about 100°.

The acidity of the curd may also be judged by the hot-iron test; an iron is heated and allowed to cool till it can just be touched with the finger; the acidity of the curd is judged by the length of the string which is formed when the iron is pressed on the curd and withdrawn.

The strength of rennet is determined by weighing out 0.5 gramme of a solid extract, or measuring 5 c.c. of a liquid extract, and diluting to 100 c.c. 1 c.c. of the solution is added to 100 c.c. of separated milk of acidity 20° at a temperature of 35° C.; the temperature is kept at 35°, and the milk slowly stirred with a thermometer till it curdles, which is indicated by the path of the thermometer becoming visible; the time which has elapsed since the addition of the rennet is noted, and the strength of the rennet calculated as parts of rennet which will cause curdling in forty minutes by the following formulæ:

$$\text{Strength} = \frac{800{,}000}{T} \text{ for solid extracts,}$$

$$\text{or} \qquad \frac{80{,}000}{T} \text{ for liquid extracts,}$$

where T = time in minutes.

The curdling should not take less than five minutes nor more than ten minutes, and should this be the case less or more of the rennet solution should be used, and the results increased or decreased proportionately.

TABLE XI

FOR CORRECTING SPECIFIC GRAVITY TO 60° F.

(*see* p. 8)

Temperature Degrees F.	Degrees of Specific Gravity observed											
	25	**26**	**27**	**28**	**29**	**30**	**31**	**32**	**33**	**34**	**35**	**36**
	Specific Gravity corrected to 60° F.											
40	23.5	24.5	25.5	26.4	27.3	28.2	29.1	30.0	31.0	31.9	32.8	33.7
42	23.6	24.6	25.6	26.5	27.5	28.4	29.3	30.2	31.1	32.0	32.9	33.9
44	23.8	24.8	25.8	26.7	27.7	28.6	29.5	30.4	31.3	32.2	33.1	34.1
46	23.9	24.9	25.9	26.8	27.8	28.7	29.6	30.5	31.4	32.4	33.3	34.3
48	24.0	25.0	26.0	26.9	27.9	28.8	29.7	30.6	31.6	32.6	33.5	34.5
50	24.1	25.1	26.1	27.0	28.0	29.0	29.9	30.9	31.8	32.8	33.7	34.7
52	24.3	25.2	26.2	27.2	28.1	29.1	30.1	31.1	32.0	33.0	33.9	34.9
54	24.5	25.4	26.4	27.4	28.4	29.3	30.3	31.3	32.3	33.3	34.2	35.1
56	24.6	25.6	26.6	27.6	28.6	29.6	30.5	31.5	32.5	33.5	34.4	35.4
58	24.8	25.8	26.8	27.8	28.8	29.8	30.8	31.7	32.7	33.7	34.7	35.7
60	**25.0**	**26.0**	**27.0**	**28.0**	**29.0**	**30.0**	**31.0**	**32.0**	**33.0**	**34.0**	**35.0**	**36.0**
62	25.2	26.2	27.3	28.3	29.3	30.3	31.3	32.3	33.3	34.3	35.3	—
64	25.4	26.5	27.5	28.5	29.5	30.5	31.5	32.6	33.6	34.6	35.6	—
66	25.6	26.7	27.7	28.7	29.8	30.8	31.8	32.9	33.9	34.9	35.9	—
68	25.9	27.0	28.0	29.0	30.1	31.1	32.1	33.2	34.2	35.2	36.2	—
70	26.1	27.2	28.2	29.2	30.3	31.3	32.4	33.4	34.5	35.5	36.5	—
72	26.4	27.4	28.4	29.5	30.5	31.6	32.6	33.7	34.7	35.8	—	—
74	26.6	27.7	28.7	29.7	30.8	31.9	32.9	34.0	35.0	36.1	—	—
76	26.9	27.9	28.9	29.9	31.0	32.2	33.3	34.4	35.4	36.5	—	—
78	27.2	28.2	29.2	30.3	31.4	32.5	33.6	34.7	35.8	36.9	—	—
80	27.4	28.4	29.5	30.6	31.7	32.8	33.9	35.0	36.1	—	—	—

TABLE XII

FOR CALCULATING SOLIDS NOT FAT FROM FAT AND SPECIFIC GRAVITY (*see* p. 40)

Correction	* Specific Gravity (Degrees)							
−1.0	25.0	25.5	26.0	26.5	27.0	27.5	28.0	28.5
*	**29.0**	**29.5**	**30.0**	**30.5**	**31.0**	**31.5**	**32.0**	**32.5**
+1.0	33.0	33.5	34.0	34.5	35.0	35.5	36.0	36.5
Fat	**Solids not Fat**							
0.0	7.40	7.50	7.65	7.75	7.90	8.00	8.15	8.25
0.25	7.45	7.55	7.70	7.80	7.95	8.05	8.20	8.30
0.5	7.50	7.60	7.75	7.85	8.00	8.10	8.25	8.35
0.75	7.55	7.65	7.80	7.90	8.05	8.15	8.30	8.40
1.0	7.60	7.70	7.85	7.95	8.10	8.20	8.35	8.45
1.25	7.65	7.75	7.90	8.00	8.15	8.25	8.40	8.50
1.5	7.70	7.80	7.95	8.05	8.20	8.30	8.45	8.55
1.75	7.75	7.85	8.00	8.10	8.25	8.35	8.50	8.60
2.0	7.80	7.90	8.05	8.15	8.30	8.40	8.55	8.65
2.75	7.85	7.95	8.10	8.20	8.35	8.45	8.60	8.70
2.5	7.90	8.00	8.15	8.25	8.40	8.50	8.65	6.75
2.75	7.95	8.05	8.20	8.30	8.45	8.55	8.70	8.80
3.0	8.00	8.10	8.25	8.35	8.50	8.60	8.75	8.85
3.25	8.05	8.15	8.30	8.40	8.55	8.65	8.80	8.90
3.5	8.10	8.20	8.35	8.45	8.60	8.70	8.85	8.95
3.75	8.15	8.25	8.40	8.50	8.65	8.75	8.90	9.00
4.0	8.20	8.30	8.45	8.55	8.70	8.80	8.95	9.05
4.25	8.25	8 35	8.50	8.60	8.75	8.85	9.00	9.10
4.5	8.30	8.40	8.55	8.65	8.80	8.90	9.05	9.15
4.75	8.35	8.45	8.60	8.70	8.85	8.95	9.10	9.20
5.0	8.40	8.50	8.65	8.75	8.90	9.00	9.15	9.25
†Change at	0.2	0.1	0.2	0.1	0.2	0.1	0.2	0.1

* The solids not fat are correct for the middle line of specific gravity; if the specific gravity falls in the top line subtract 1 from the solids not fat; thus 26.0 sp. gr. and 3.0% fat give 7.25% solids not fat; if the specific gravity falls in the bottom line, add 1 to the solids not fat; thus 34.0 sp. gr. and 3.0% fat give 9.25% solids not fat.

† The last line indicates where the change of solids not fat takes place; in a column with 0.2 at the bottom use the column itself for the percentage of fat given, and 0.05, 0.10, and 0.15 above, but use the figure immediately below for 0.2 above; thus 30.0 sp. gr. and 3.1% fat give 8.25% solids not fat, but 30.0 sp. gr. and 3.2% fat give 8.30% solids not fat; in a column with 0.1 at the bottom, use the column itself for the percentage of fat given and 0.05 above, but change to the figure immediately below for 0.1 or more above; thus 30.5 sp. gr. and 3.30% fat give 8.40% solids not fat, but 30.5 sp. gr. and 3.40% fat give 8.45% solids not fat.

TABLE XIII

FOR CALCULATING FAT FROM TOTAL SOLIDS AND SPECIFIC GRAVITY (*see* p. 40)

Specific Gravity Observed										
	A25.0 B29.0 C33.0	25.5 29.5 33.5	26.0 30.0 34.0	26.5 30.5 34.5		A27.0 B31.0 C35.0	27.5 31.5 35.5	28.0 32.0 36.0	28.5 32.5 36.5	
Total Solids A	Fat				Total Solids B	Fat				Total Solids C
7.00	0.50	0.40	0.30	0.20	8.0	0.10	—	—	—	9.00
7.25	0.70	0.60	0.50	0.40	8.25	0.30	0.20	0.10	—	9.25
7.50	0.90	0.80	0.70	0.60	8.5	0.50	0.40	0.30	0.20	9.50
7.75	1.15	1.05	0.90	0.80	8.75	0.70	0.60	0.50	0.40	9.75
8.00	1.35	1.25	1.15	1.05	9.00	0.90	0.80	0.70	0 60	10.00
8.25	1.55	1.45	1.35	1.25	9.25	1.15	1.05	0.90	0.80	10.25
8.50	1.75	1.65	1.55	1.45	9.50	1.35	1.25	1.15	1.05	10.50
8.75	1.95	1.85	1.75	1.65	9.75	1.55	1.45	1.35	1.25	10.75
9.00	2.15	2.05	1.95	1.85	10.00	1.75	1.65	1.55	1.45	11.00
9.25	2.40	2.30	2.15	2.05	10.25	1.95	1.85	1.75	1.65	11.25
9.50	2.60	2.50	2.40	2.30	10.50	2.15	2.05	1.95	1.85	11.50
9.75	2.80	2.70	2.60	2.50	10.75	2.40	2.30	2.15	2.01	11.75
10.00	3.00	2.90	2.80	2.70	11.00	2.60	2.50	2.40	2.30	12.00
10.25	3.20	3.10	3.00	2.90	11.25	2.80	2.70	2.60	2.50	12.25
10.50	3.40	3.30	3.20	3.10	11.50	3.00	2.90	2.80	2.70	12.50
10.75	3.65	3.55	3.40	3.30	11.75	3.20	3.10	3.00	2.90	12.75
11.00	3.85	3.57	3.65	3.55	12.00	3.40	3.30	3.20	3.10	13.00
11.25	4.05	3.95	3.85	3.75	12.25	3.65	3.55	3.40	3.30	13.25
11.50	4.25	4.15	4.05	3 95	12.50	3.85	3.75	3.65	3.55	13.50
11.75	4.45	4.35	4.25	4.15	12.75	4.05	3.95	3.85	3.75	13.75
12.00	4.65	4.55	4.45	4.35	13.00	4.25	4.15	4.05	3.95	14.00
12.25	4.90	4.80	4.65	4.55	13.25	4.45	4.35	4.25	4.15	14.25
12.50	5.10	5.00	4.90	4.80	13.50	4.65	4.55	4.45	4.35	14.50
12.75	5.30	5.20	5.10	5.00	13.75	4.90	4.80	4.65	4.55	14.75
13.00	5.50	5.40	5.30	5.20	14.00	5.10	5.00	4.90	4.80	15.00

If the total solids do not exactly agree with a figure in the Table add the excess of total solids to the fat; thus, given 30.5 sp. gr. and 11.8% total solids, add 0.05 to fat corresponding with 11.75% total solids = 3.35% fat.

If the specific gravity lies in line marked A use the total solids column marked A; if in B use total solids column B; if in C use total solids column C.

APPENDIX

PREPARATION OF STANDARD SOLUTIONS

As the preparation of a standard solution is rarely, if ever, undertaken while an analysis is being performed, and as also it is tedious to wade through a description of a method when seeking only for the quantities to be weighed or measured for the solution, details of the preparation have been removed from the text; the arrangement is alphabetical.

Alcoholic Soda Solution.—$\frac{N}{2}$ approximately. Add 25 c.c. of a 50 per cent. caustic soda solution (*see* p. 93) to 1 litre of alcohol (sp. gr. 0.830) and mix well; allow the solution to stand a day, filter into a dry stoppered bottle, and titrate (*see* Titration) against $\frac{N}{2}$ hydrochloric acid (*see* p. 92) to ascertain strength. As this solution loses strength on keeping, it must be titrated each time it is used.

Amyl Alcohol (for Gerber test) should conform to the following: Density, 0.8145 to 0.816 at 15.5° C. (Water at 15.5° C. = 1.) Boiling-point: should not begin to boil below 124° C.; not more than 5 c.c. should distil at 127.5° C. when 25 c.c. are boiled in a 100 c.c. flask, and the boiling-point should not rise above 130.5° C.; 10 c.c. should completely mix with 10 c.c. of hydrochloric acid (sp. gr. 1.17) and the addition of 1.5 c.c. of water should produce a permanent turbidity. 2 c.c. treated in the Gerber bottle with 10 c.c. water and 10 c.c. sulphuric acid should yield no layer of "fat."

Barium Chloride Solution, $\frac{N}{5}$.—24.45 grammes of crystallised barium chloride are dissolved in 1 litre of distilled water.

Baryta Solution.—Dissolve 33–35 grammes of barium hydrate in 2 litres of water; allow to settle, and store in a bottle fitted with a soda lime tube to prevent access of CO_2. Check the strength by titration (*see* p. 94).

Caustic Soda. *See* Soda.

Fehling's Solution.—Copper sulphate solution: Dissolve 34.639 grammes of crystallised copper sulphate in water, and make up to 500 c.c. Alkaline tartrate solution: Dissolve 173 grammes of pure sodium potassium tartrate (Rochelle Salt), and 51 to 55 grammes of sodium hydroxide of good quality in water, and make up to 500 c.c.

Hydrochloric Acid $\frac{N}{2}$.—Dilute 50 c.c. strong hydrochloric acid to 1 litre; ignite a quantity of pure sodium bicarbonate at a dull red heat, and cool in a desiccator; weigh (accurately to 1 milligramme) portions of this, of about 1 gramme each, dissolve in water, add a little methyl orange, and run in the hydrochloric acid solution from a burette till the sodium carbonate solution turns pink; the solution should be covered as much as possible with a watch-glass to prevent loss by spirting as the CO_2 is given off. Wash the watch-glass into the solution, and boil it, and continue the titration till the pink colour is permanent on boiling. It is well to place a beaker containing an equal bulk of distilled water, and the same volume of methyl orange solution, and one drop (0.05 c.c.) of hydrochloric acid, and titrate till the sodium carbonate solution has the same colour, and subtract 0.05 c.c. from the quantity of acid used. If the solution is $\frac{N}{2}$, 20 c.c. should be required for each 0.53 grammes of sodium carbonate; if this quantity is not used dilute in the proportion of the quantity used to 20 c.c., or use the solution as it is, and multiply by 20 divided by the quantity used to obtain its value in terms of $\frac{N}{2}$ acid. $\frac{N}{10}$ solution is made from $\frac{N}{2}$ solution by diluting 200 c.c. to 1 litre.

Iodine Solution (Wijs).—Dissolve 13 grammes of iodine in 1 litre of 95 per cent. acetic acid (but made by diluting 100 per cent. acid with 5 per cent. of distilled water); pass in a current of chlorine till the litre of the solution has been doubled; this point is indicated by a change of colour.

Magnesia Mixture.—Mix 60 grammes magnesium chloride ($MgCl_26OH_2$), 145 grammes ammonium chloride, 600 c.c. water, and 300 c.c. ammonia solution (sp. gr. 0.880).

Mercuric Nitrate Solution (Wiley).—Dissolve mercury in twice its weight of nitric acid (sp. gr. 1.42), and, after solution, add an equal bulk of water.

Nessler Solution.—Dissolve 35 grammes of potassium iodide in 100 c.c. of water; set aside a few c.c. of this solution, and add to the remainder a saturated solution of mercuric chloride till a permanent red precipitate is formed; add the small portion set aside, and cautiously drop in mercuric chloride solution, till a faint permanent precipitate is left. Dissolve 160 grammes of potassium hydroxide in water; *cool* the solution, and add it to the potassium mercuric iodide solution. Make up to 1 litre, add a little mercuric chloride solution and allow to stand till clear.

Phenolphthalein Solution.—Dissolve 5 grammes of phenolphthalein in 600 c.c. of alcohol and dilute to 1 litre with water. Methylated spirit may be used, and if necessary the solution should be heated with animal charcoal, and filtered till clear.

Silver Nitrate, $\frac{N}{10}$.—Dissolve 17.000 grammes of silver nitrate in 1 litre of water.

Soda Solution, 50 per cent.—Dissolve 250 grammes of caustic soda (purified by alcohol) in 250 c.c. of water; allow to stand till clear, and store in the apparatus described on p. 68.

For Kjeldahl Process.—Dissolve 300 grammes of caustic soda in water and make up to 1 litre. After standing a few days, filter through glass wool.

$\frac{N}{2}$.—Dilute 25 c.c. of the 50 per cent. solution to 1 litre. Check the strength by titration (*see below*).

$\frac{N}{10}$.—Dilute 5 c.c. of the 50 per cent. solution to 1 litre. Check the strength by titration.

Sodium Hypobromite Solution.—Add 1 c.c. of bromine to 10 c.c. of soda solution (for Kjeldahl process).

Strontia Solution.—Dissolve 28–30 grammes of strontium hydrate in 2 litres of water. Treat as baryta solution (p. 92).

Sulphuric Acid. *For Gerber Process.*—Should contain 90–91 per cent. H_2SO_4. Acid of specific gravity 1.820 to 1.825 fulfils this requirement.

For Reichert Process.—Dilute 25 c.c. strong sulphuric acid to 1 litre of water; 2 c.c. of 50 per cent. soda solution should neutralise about 35 c.c. of this solution; adjust the strength, if necessary, by adding acid or water.

$\frac{N}{10}$.—Dilute 3 c.c. of strong sulphuric acid to 1 litre. Check the strength as for hydrochloric acid (0.2 grammes only of the sodium carbonate is weighed for $\frac{N}{10}$ acid, and 100 c.c. of $\frac{N}{10}$ acid should be required for 0.53 grammes sodium carbonate).

Thiosulphate Solution.—Dissolve 25 grammes of pure sodium thiosulphate and 1 gramme of salicylic acid in 1 litre of water. Allow to stand for a few days and filter. Weigh accurately about 0.25 gramme of iodine in a small stoppered flask, add 2 grammes potassium iodide and 2 c.c. of water, and shake gently till the iodine is dissolved. Dilute with water and transfer to a larger vessel, and run in the thiosulphate solution from a burette till the yellow colour just disappears. Repeat this two or three times, and from the mean results calculate the weight of iodine equivalent to 1 c.c.

Titration.—Take two burettes, fill one with the alkaline solution, and the other with hydrochloric acid

solution of corresponding strength. Measure 25 c.c. of the acid solution, add a few drops of phenolphthalein solution (or cochineal solution if this is used in the experiment for which the alkali is to be employed), and run in the alkali till a pink colour (violet with cochineal) is produced. Note the volume of alkali used. Repeat this experiment two or three times, and from the mean of the results calculate the ratio of the alkali solution to the acid solution. Thus, if 24.2, 24.15, and 24.2 c.c. of $\frac{N}{2}$ soda were used for 25 c.c. acid the ratio is

$$\frac{25}{24.17} = 1.034 \text{ or } 1 \text{ c.c. of soda} = 1.034 \text{ c.c. acid.}$$

If the acid is strictly $\frac{N}{2}$ the soda is $\frac{N}{2} \times 1.034$; if the acid is, for instance, $\frac{N}{2} \times 1.011$, the soda is then $\frac{N}{2} \times 1.034 \times 1.011 = \frac{N}{2} \times 1.0454$.

The soda may be diluted in the ratio of 1000 parts to 1045.4 parts, *i.e.* 45.4 c.c. of water are added to each litre, but it is preferable to use the solution as it is, and multiply the results by the factor.

To standardise $\frac{N}{10}$ solutions ($\frac{N}{11}$ strontia) 5 c.c. of the $\frac{N}{2}$ hydrochloric acid should be carefully measured, a few drops of phenolphthalein solution added, and the alkali run in till a faint pink colour is seen. Several titrations should be made, and the mean taken. This figure divided into 2.5 will give the strength of the solution in terms of normal. Thus if 26.2, 26.3, and 26.25 c.c. were used in three experiments the mean is 26.25, and the strength is $\frac{2.5}{26.25} = 0.09505$ N.

INDEX

BALLANTYNE & COMPANY LTD
Tavistock Street Covent Garden
London

BAIRD & TATLOCK (LONDON) LTD.'S LIST OF APPARATUS AND CHEMICALS REQUIRED FOR MILK ANALYSIS, &c.

As mentioned in the "Laboratory Book of Dairy Analysis," by H. DROOP RICHMOND, F.I.C.

ALL the apparatus detailed below are required for a full equipment; the quantity will vary according to requirements:

THE ANALYSIS OF MILK

Sampling

Page	*Item No.*	
5	1	Sample bottles, oval shape, with corks, 5 oz.
6	2	Beakers, with spout, No. 5.
6	3	Brush, fine wire, for mixing.

Specific Gravity

6	4	Lactometer, ordinary pattern, paper scale.
7	5	Lactometer, Veith's pattern, 25–35.
7	6	Lactometer, Soxhlet's pattern, about 13 in. long.
7	7	Lactometer jar, zinc, 4 in. by $1\frac{3}{4}$ in., with spout.
8	8	Thermo-lactometer, Quevenne's.
10	9	Beaker flasks, 200 c.c.
10	10	Sprengel tube, 10 c.c.

Estimation of Total Solids

11	11	Platinum basin, flat, shallow, with spout, $2\frac{3}{4}$ in.
11	12	Porcelain capsule, glazed all over. Tantalum basin. Silica basin.
11	13	Pipette for milk, 5 c.c., with mark.
11	14	Pipette to hold 5 grammes of milk.
11	15	Water-bath to take 6 porcelain capsules.
12	16	Desiccator, Scheibler's pattern, 6 in.
12	17	Balance, Bunge's 200 grammes, sensibility to $\frac{1}{10}$th mgm. on plate-glass sole.
12	18	Set of gilt weights for above, 50 to .001 grammes.

Page	*Item No.*	
		Estimation of Ash, &c.
12	19	Muffle furnace, No. 461.
12	20	Fireclay muffle for ditto.
12	21	Platinum wire, 6 in. long.
12	22	Tripod, triangular, 8 in. high.
12	23	Bunsen burner, $\frac{1}{2}$ in. Argand burner.
12	24	India-rubber tubing for above, heavy walls.
12	25	Pipe-clay triangle, Clowes' improved form. Triangle silica.
13	26	Filter-paper, C.S. and S. 589, black band.
15	27	Porcelain basin, round-bottomed, with spout, diameter $3\frac{1}{2}$ in.
		Estimation of Acidity
16	28	Acidimeter for testing acidity, consisting of special burette, stand, and dropping bottle.
		Estimation of Fat
17	29	Gerber's butyrometer for 4 samples, with accessories as follows: 4 bottles; 1 pipette, 3 c.c. for cream; 1 „ 10 c.c. 100 divs. for water; 1 „ 11 c.c. for milk; 1 „ 10 c.c. for acid; 1 „ 1 c.c. for amyl alcohol.
17	30	Leffman-Beam centrifugal machine, for 4 samples, with accessories as follows: 4 bottles; 1 pipette, 3 c.c.; 1 „ 9 c.c. 1 „ 15 c.c.
20	31	Richmond's shaking stand for 8 Gerber's tubes.
25	32	Pair of tared tin dishes, 2 in. by $1\frac{5}{8}$ in. for weighing cream.
		Gravimetric Estimation of Fat
29	34	Werner-Schmid tube.
28	35	Fat extraction thimble.
28	36	Glass mortar, 3 in. diameter, with pestle.

Page	Item No.	
35	37	Funnel, 2½ in.
35	38	Filter-paper, C.S. and S 595, to suit.
35	39	Beakers, No. 3, with spout.
35	40	6 glass stirring rods, 6 in. long.
35	41	Conical flasks, 6 oz.
	41*a*	Stoppered cylinders.
	41*b*	Wash-bottle tubes.
29	42	Soxhlet's Fat Extraction Apparatus as follows: 1 water-bath for 3 apparatus on stand; 1 set Bunsen's for ditto; 3 fat extractors, 60 grammes; 3 flasks with short necks, 4 oz.; 3 condensers with india-rubber caps; 1 stand with three-armed clamp for supporting condensers.
30	43	Stokes tube.
30	44	Burette, 100 c.c. $\frac{1}{10}$ths, with stopcock. Mahogany stand for ditto.

Estimation of Milk Sugar

32	45	Mitscherlich's polariscope, with 100 and 200 mm. tube and burner.
32	46	Hard glass tubes, 10 × 1 c.m., with one end drawn down.
31	47	Flasks, with mark 100 c.c., unstoppered.
33	48	Policemen, with india-rubber tops, for stirring.
33	49	Filter pump glass.
33	50	Glass tubing, assorted.
33	51	Wash-bottles, fitted with cork and tubes, 1 litre.

Estimation of Proteins

34	52	Gooch's porcelain crucible.
34	53	Asbestos fibre, for use with above.

Estimation of Casein and Albumin

35	54	Porcelain crucibles, No. 1, with lids.
35	55	Crucible tongs, gun-metal, with platinum tips.

Estimation of Nitrogen

35	56	Kjeldahl's apparatus, with litre copper flask, Liebig's condenser, &c.
35	57	Flask, 200 c.c., hard glass, round bottom.

Page	Item No.	
37	58	Burette, fitted with soda lime tube, and bottle with
39		soda lime tube to hold strontia solution.
40	58a	Richmond's improved milk slide rule.
42	58b	Collins' milk scale.

Analysis of Liquid Milk-Products

47	59	Pipette, 5 c.c., short form with india-rubber tube at top.
48	60	Flask, with side tube, 250 c.c.
48	61	Liebig's condenser, for use with ditto.
39	61a	Lobeck's catalase tubes.
49	62	Nessler tubes, marked at 50 c.c.

The Application of Analysis to the Solutions of Problems

54	62a	Separating funnel for salicylic and benzoic acid.
54	63	Test-tubes, 6 $\times \frac{5}{8}$ in.
56	64	High-speed centrifuge.
58	65	Microscope, with $\frac{2}{3}$ in. and $\frac{1}{6}$ in. objective, one eyepiece and substage condenser and polariscope fitted.
58	65a	Test-tubes, 2 $\times \frac{1}{4}$ in.
14, 64, 94	66	2 burettes, 50 c.c. $\frac{1}{10}$ths.
18, 64, 94	67	Stand, double, for ditto.
14, 64, 95	68	6 dropping bottles, with grooved stopper, 30 c.c.

THE ANALYSIS OF BUTTER

Estimation of Water, &c.

64	69	Sand-bath, 6 in.
64	70	1 round tripod for ditto.
64	71	1 Bunsen burner for ditto.
64	72	Porcelain basins, No. 1.
64	73	Glass rod for ditto.
66	74	Glass plates, $2\frac{1}{2} \times 2\frac{1}{2}$ in.
66	75	Set of pipettes, with mark 5, 10, 20, and 25 c.c.

Page	*Item No.*	
		Estimation of the Volatile Fatty Acids
69	76	Caustic soda apparatus.
70	77	Polenske apparatus.
71	78	Flask, CO_2, 6 oz.
71	79	Liebig's condenser for ditto.
71	80	Porcelain basin, 6 in. diameter.
74	81	Microscope slides, 3 × 1 in.
74	82	Microscope cover glasses, $\frac{5}{8}$ in. square, No. 2.
75	83	Bottles, 4 oz. N.M., flat-stoppered.
		Determination of Refractive Index
76	84	Zeiss' butyro-refractometer.
		THE ANALYSIS OF CHEESE
83	85	Porcelain capsules, glazed all over.
84	86	Flask, 250 c.c. with mark, stoppered.
84	87	Separators, 500 c.c. cylindrical, with stopper and stopcock.
86	88	Hot-air bath, 7 × 7 in., on stand.
86	89	Thermometer, engraved up to 110° C.
86	90	Incubator, B. and T.'s form, slag wool lined, with capsule regulating at 37° C., complete with thermometer.
86	91	Pair watch-glasses with binder.
86	92	100 c.c. flask with mark.

CHEMICALS AND STANDARD SOLUTIONS

THE quantities detailed below are necessary for a large laboratory. They may be reduced in proportion when only a small outfit is required :

5 lb. acetic acid 100%.
1 lb. acetone.
4 oz. phenolphthalein indicator, 0.5%
1 litre sulphuric acid $\frac{N}{10}$ solution.
4 oz. potassium chromate indicator.
1 litre silver nitrate $\frac{N}{10}$ solution.
1 w. qt. hydrochloric acid, pure.

1 w. qt. ammonia, .880.
2 lb. Schering's formalin.
½ lb. ammonium oxalate, pure.
½ lb. ammonium carbonate, pure.
½ lb. magnesia mixture.
1 litre caustic soda $\frac{N}{10}$ solution.
1 lb. Kieselguhr special for filtering.
1 w. qt. amylic alcohol } for Leffmann–Beam or Gerber
1 carboy sulphuric acid } process.
1 lb. soda lime.
1 w. qt. alcohol.
1 w. qt. petroleum ether.
5 lb. caustic soda, pure.
1 w. qt. sulphuric acid free N.
1 lb. mercury redistilled. } for Kjeldahl's
2 lb. potassium bisulphate. } process.
1 lb. sodium sulphide pure reagent.
4 oz. cochineal indicator.
1 lb. phosphotungstic acid.
2 lb. magnesium sulphate, pure.
1 w. qt. nitric acid, pure.
2 lb. sodium carbonate.
1 lb. barium chloride.
1 w. qt. ether .720 meth.
1 tube rennet tablets.
1 lb. beeswax.
1 roll each blue and red litmus paper and turmeric paper.
½ lb. ferric chloride.
1 oz. para-phenylenediamine.
1 oz. ortol.
1 oz. resorcine.
2 lb. sodium bicarbonate.
½ lb. potassium ferrocyanide, pure.
½ lb. iodine re-sublimed.
1 lb. potassium iodide.
2 lb. hydrogen peroxide, 20 vol.
2 oz. picric acid.
½ oz. silver nitrate.
1 lb. calcium chloride, dry.
1 lb. chloroform.
2 lb. Rochelle salt.

1 oz. furfural.
2 lb. copper sulphate.
1 lb. mercuric chloride.
1 lb. magnesium chloride.
5 lb. caustic potash.
¼ lb. strontium hydrate.
1 lb. sodium thiosulphate.
1 oz. salicylic acid.
½ litre Nessler's reagent.
2 lb. glycerine.
½ lb. bromine.
½ lb. pumice.
